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(54) INTERMEDIATE FILM FOR LAMINATED GLASS AND LAMINATED GLASS

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a laminated glass which maintains basic performance necessary for a laminated glass and little efflorescence in the peripheral part even in a high humidity atmosphere by forming an intermediate film comprising a plasticized polyvinyl acetal resin film having a specified or lower haze when the film is immersed in water.

SOLUTION: When the intermediate film for a laminated glass comprising a plasticized polyvinylacetal resin film having 0.3 to 0.8 mm thickness is immersed in water at 23° C, efflorescence of the film after 24 hours is controlled to ≤ 50 % haze. To obtain the haze value above described, sodium salts and potassium salts which cause the efflorescence and which are mixed from the source material are controlled to have ≤ 10 μ m particle size and to give ≤ 50 ppm sodium concn. and ≤ 100 ppm potassium concn. in the intermediate film. To effectively prevent efflorescence at high humidity, compds. which can form complexes with sodium salts and potassium salts, and org. acids and amines compatible with the resin and the plasticizer are preferably added as dispersants.

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AIMS

aim(e)11

aim 11]An interlayer for glass laminates when it is an interlayer for glass laminates which consists of plasticized polyvinyl polyvinylacetate resin films and said 0.3-0.8-mm-thick interlayer is immersed in ** water, wherein Hayes of 24 hours after is 50% or less.

aim 21]The interlayer for glass laminates according to claim 1 whose particle diameter of sodium : in an interlayer is 10 micrometers or less.

aim 31]The interlayer for glass laminates according to claim 1 or 2 whose particle diameter of :ium salt in an interlayer is 5 micrometers or less.

aim 41]The interlayer for glass laminates according to claim 1, 2, or 3 whose sodium concentration : in an interlayer is 50 ppm or less.

aim 51]The interlayer for glass laminates according to claim 1 whose particle diameter of potassium : in an interlayer is 10 micrometers or less.

aim 61]The interlayer for glass laminates according to claim 1 or 5 whose particle diameter of :assium salt in an interlayer is 5 micrometers or less.

aim 71]The interlayer for glass laminates according to claim 1, 5, or 6 whose potassium :centration in an interlayer is 100 ppm or less.

aim 81]The interlayer for glass laminates according to claim 1, 2, 3, 4, 5, 6, or 7 which is a thing :aining a compound which can form sodium salt and potassium salt, and a complex.

aim 91]The interlayer for glass laminates according to claim 1, 2, 3, 4, 5, 6, or 7 which is a thing :aining amine which is compatible in organic acid which is compatible in resin and a plasticizer, :in, and a plasticizer.

aim 101]The interlayer for glass laminates according to claim 1, 2, 3, 4, 5, 6, 7, 8, or 9 which is a :ng containing at least one sort chosen from a group which consists of alkali metal salt and alkaline :th metal salt.

aim 111]The interlayer for glass laminates according to claim 10 whose alkali metal salt is a thing :n a particle diameter of 3 micrometers or less and whose alkaline earth metal salt is a thing with a :ticle diameter of 3 micrometers or less.

aim 121]The interlayer for glass laminates according to claim 10 or 11 whose alkali metal salt is the :li metal salt of organic acid of the carbon numbers 5-16 and whose alkaline earth metal salt is the :aine earth metal salt of organic acid of the carbon numbers 5-16.

aim 131A]A glass laminate characterized by making the interlayer for glass laminates according to :m 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, or 12 come to intervene between glass of a couple at least.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[01]

[Detailed Description of the Invention] This invention relates to the glass laminate which used the interlayer for glass laminates, and the above-mentioned interlayer for glass laminates.

[02]

[Detailed Description of the Prior Art] The glass laminate with which it comes conventionally to fasten the interlayer which consists of a plasticized polyvinyl butyral between the glass plates of at least two sheets, it has basic performance transparency, weatherability, and an adhesive property are good, and penetration resistance moreover and required for the glass laminate of a glass fragment not separating easily, for example, is widely used for the winduppane of a car or a building.

[03] This kind of glass laminate is inferior in moisture resistance, although the above-mentioned adhesive performance is good and excellent in safety. That is, by the periphery of a glass laminate, when above-mentioned glass laminate is placed into a humid atmosphere, since the interlayer touches environmental air directly, the problem which the interlayer of a periphery milks arises.

[04] The additive agent for performing adhesive strength adjustment of an interlayer and glass described below is participating in this abstrusism. In order to demonstrate the function as the above-mentioned glass laminate enough, it is required to adjust the adhesive strength of an interlayer and glass so that it may fall within a proper range. Namely, if the danger of the glass fragment damaged by a shock from the outside, etc. separating and dispersing from an interlayer, and doing injury to a human body etc. if the adhesive strength of an interlayer and glass is too weak becomes high and the adhesive strength of an interlayer and glass is too strong conversely, Glass and an interlayer are utteraneously damaged by the shock from the outside, etc., and the danger of the adhesion fragment glass and an interlayer dispersing and doing an obstacle to a human body etc. becomes high.

[05] On the other hand, when there is adhesive strength of an interlayer and glass within proper limits. Since the phenomenon in which partial interfacial peeling of an interlayer and glass happens, in an interlayer extends arises at the same time glass is damaged while glass breakage reaches far wide and happens, the impact-absorbing effect and a penetration preventive effect become large.

[06] Therefore, in case of the accident of transport-airplane machines, such as a car, in order to avert a shock in case a driver and a passenger collide to glass or to prevent penetration, in case of accident of a building, in order to prevent the missile from the outside from penetrating glass or prevent scattering of a glass fragment, it is required to adjust the adhesive strength of an interlayer and glass so that it may fall within a range proper like ***.

[07] In order to adjust the adhesive strength of an interlayer and glass within proper limits conventionally in view of the above, the adhesive strength regulator for interlayers has been mined variously. In JP 46-4270B, the interlayer for glass laminates which consists of a polyvinyl ester resin composition which carries out specific amount content of 0.2 to 0.8 % of the weight of the resin and the metal alkyl carboxylate specific as an adhesive strength regulator is proposed. The interlayer by the above-mentioned proposal tends to adjust the adhesive strength of an interlayer glass to the proper range by changing the distribution quantity of metal alkyl carboxylate in an interlayer part and an interlayer inner layer part, or changing the moisture content in an interlayer.

[08] However, the interlayer containing metal alkyl carboxylate like the above-mentioned proposal,

Since the interlayer is carrying out direct contact to air in the periphery of the glass laminate if the glass laminate which moisture resistance fell and was manufactured using this interlayer is neglected under a humid atmosphere, While the quantity of metal alkyl carboxylate increases, there is a problem that the abstrusism by moisture absorption of an interlayer happens violently. Although it can prevent whether the abstrusism of the above-mentioned interlayer reduces the quantity of metal alkyl carboxylate as much as possible, and by losing, in that case, the fatal problem as a glass laminate of the adhesive strength of an interlayer and glass becoming stronger than the proper range too much, and glass and an interlayer being simultaneously damaged by the shock from the outside, etc., or becoming easy to penetrate had occurred.

[0009] In JP 44-32185B, contain 0.1 to 0.8% of moisture, and The monocarboxylic acid of six to 22 carbon atom, The dicarboxylic acid of four to 12 carbon atom, and the aliphatic series monodiamino monocarboxylic acid of two to 6 carbon atom, The interlayer for glass laminates which carries out 0.01-3 weight-section content of the aliphatic series monodiamino dicarboxylic acid of four to 5 carbon atom and at least one organic acid chosen from citrate and these mixtures per resin 100 weight section and which consists of shaping polyvinyl-acetal resin is proposed.

[0010] However, when carboxylic acid is added, there is a problem that adhesiva strength changes with time. The problem that the heat resistance and the weatherability of an interlayer fall under the influence of acid arises.

[0011] In the glass which pasted the glass of at least two sheets together with the plasticized-polyvinyl-acetal-resin constituent in JP 48-5772B, The glass laminate making sodium metal salt of the aliphatic carboxylic acid of the carbon numbers 10-22 contain in this plasticized-polyvinyl-acetal-resin constituent is proposed.

[0012] In JP 53-18207B, using the alkali metal salt or alkaline earth metal salt of monocarboxylic acid or dicarboxylic acid is proposed as an adhesive strength regulator in the plasticized-polyvinyl-acetal-resin interlayer of a glass laminate.

[0013] In the two above-mentioned proposals, each uses metal salt of carboxylic acid with a comparatively large carbon number as an adhesive strength regulator from it being easy to dissolve in the plasticizer contained in an interlayer.

[0014] However, when metal salt of carboxylic acid with a large carbon number is used as an adhesive strength regulator, there is a problem that the adhesive strength of an interlayer and glass changes with time progress (temporality). That is, even if early adhesive strength is proper, adhesive strength declines gradually with temporality, and when shocked, glass exfoliates easily. In order to prevent this adhesive strength fall, it is necessary to keep an interlayer for one to two months under 40-50 °C atmosphere, and to ripe, and. Since an interlayer has adhesiveness, autohesion nature, etc., even if keeping it under the above atmosphere for a long period of time ripes difficult actually, although the fall of adhesive strength with the passage of time can be controlled, it cannot be made for there to be nothing, but the above-mentioned problem will still remain.

[0015] In JP 60-210551A, to plasticized-polyvinyl-acetal-resin 100 weight section, The glass laminate with which the glass of at least two sheets sticks, and it comes to unite it by whether 0.02 to potassium monocarboxylate 0-40 weight section and 0.01 to modified silicone oil 0.20 weight section whose carbon numbers are 1-6 contain, and the interlayer to which it adheres is indicated. However, since this metal salt solidified in particle state in the interlayer depending on the kind of metal salt used and this glass laminate caused a white bluish mark, the perfect thing was not able to say from a viewpoint of preventing the white bluish mark by long-term moisture absorption.

[0016] In JP 2-41547B, the polyvinyl-butyl sheet which uses alkali or alkaline-earth-metal-formate for an adhesive strength regulator is proposed. In the Patent Publication Heisei No. 502594 [six to] gazette, the interlayer which added potassium acetate as an adhesive strength regulator in the example is used.

[0017] In the three above-mentioned proposals, in order to cancel said problem in the case of using metal salt of carboxylic acid with a large carbon number as an adhesive strength regulator, metal salt of carboxylic acid with a comparatively small carbon number is used.

[0018] However, if metal salt of carboxylic acid with a small carbon number is used as an adhesive strength regulator, Although the problem of an adhesive strength fall with the passage of time of an interlayer and glass is canceled, the moisture resistance of an interlayer becomes insufficient and, as a result, another problem of becoming easy to start the abstrusism by moisture absorption to the edge

t (end) of a glass laminate occurs.

19]That is, since hygroscopicity is under the usual atmosphere (humidity), when processing it into glass laminate, it is common [an interlayer] to process it by controlling the humidity and doubling, example so that water content may be about 0.5 or less % of the weight under the atmosphere of RH. However, since the edge part of a glass laminate is in an unreserved state, under a high humidity atmosphere, an interlayer absorbs moisture and water content usually rises to about 2 to 3% weight. At this time, water gathers for the circumference of metal salt of carboxylic acid with a carbon number of potassium acetate, magnesium acetate or potassium formate etc., which acts as a minute crystal in an interlayer, and albinism is caused. If the addition of carboxylic acid in a small carbon number or its salt is decreased in order to reduce albinism, the adhesive strength in interlayer and glass will deviate from the proper range, and will become insufficient [impact-option nature, penetration resistance, etc. of a glass laminate].

20]As a trial to improve, the white bluish mark of the interlayer containing carboxylic acid metal salt in JP 5-188250.A. The interlayer for glass laminates currently formed from polyvinyl-acetal resin, plasticizer, and the resin composition in which aliphatic series mono- or di-carboxylic acid is posed.

21]The interlayer for glass laminates which becomes JP 7-41340.A from the resin composition containing polyvinyl-acetal resin, a plasticizer, carboxylic acid metal salt, and straight-chain fatty acid exposed.

22]However, the glass laminate using the interlayer for glass laminates of the above-mentioned proposal is not still enough, although the white bluish mark of the edge part after a humidity resistance test is reduced. And when it is going to reduce a white bluish mark further and the content of straight-chain fatty acid is increased, and a glass laminate is placed under relatively high temperature, there is a possibility of producing foaming and discoloration.

23]Although the interlayer of the above-mentioned proposal tries solution of a white bluish mark improvement of an adhesive strength regulator, also in the interlayer which is not adding the adhesive strength regulator, the white bluish mark by moisture absorption produces it. It became clear our latest research that the inquiry in the resin described below is involving as one of the cause is.

24]Although the interlayer for glass laminates of this invention uses polyvinyl-acetal resin as the ingredients, the neutralization process is included when manufacturing polyvinyl-acetal resin. In the solution of sodium salt, such as sodium hydroxide and sodium bicarbonate, is used in this neutralization process, for example, when those sodium salt is used superfluously and new sodium salt generates by neutralization, sodium salt remains in the polyvinyl-acetal resin obtained.

25]In the interlayer for glass laminates of this invention, a white bluish mark by moisture absorption of the interlayer for glass laminates obtained in order to promote condensation of water, when it becomes little state at the time of a polymerization and desiccation and polyvinyl-acetal resin absorbs water. Sodium salt may remain also to polyvinyl alcohol and this sodium salt may cause a white bluish mark by moisture absorption of the interlayer for glass laminates.

26]In recent years, the motion which uses a glass laminate for side glasses and the various things of a car prospers, in these uses, also when using the periphery of a glass laminate in the reserved state, it is increasing, and the request to albinism prevention is increasingly stronger.

27]The place which this invention solves the above-mentioned problem and is made into the purpose, without spoiling basic performance required for glass laminates, such as transparency, weatherability, an adhesive property, and penetration resistance, even when moreover placed into a humid atmosphere, there is a white bluish mark of a glass laminate edge part in providing the interlayer for [little] glass laminates, and the glass laminate is it.

28]This invention is an interlayer for glass laminates which consists of a polyvinyl alcohol test-method. As the above-mentioned aldehyde, aldehyde of the carbon numbers 3-10 is preferred. If 10 is exceeded, the reactivity of acetalization will fall, and moreover it will become easy to generate a block of resin during a reaction, and will become easy to follow difficulty on composition of resin.

[0028]When an interlayer for glass laminates of this invention immerses a 0.3-0.8-mm-thick interlayer in 23 ° water, Hayes of 24 hours after is 50% or less.

[0029]When this invention persons immerse a 0.3-0.8-mm-thick interlayer in 23 ° water. Even when Hayes of 24 hours after was placed into atmosphere where 50% or less of interlayer for glass laminates is humid, there were few white bluish marks of a glass laminate edge part, they found out excellent in moisture resistance, and completed this invention.

[0030]Since it becomes insufficient to prevent a white bluish mark under high humidity and it is inferior to moisture resistance when above-mentioned Hayes exceeds 50%, it is limited to a mentioned range. Above-mentioned Hayes as used herein shall mean a value which used and measured an integral equation turbidity meter 24 hours afterward, when a 0.3-0.8-mm-thick interlayer is immersed in 23 ° water.

[0031]An interlayer for glass laminates of this invention consists of a plasticity polyvinyl-acetal resin layer, and the above-mentioned plasticity polyvinyl-acetal resin layer uses polyvinyl-acetal resin as the main ingredients.

[0032]As the above-mentioned polyvinyl-acetal resin, average degree-of-acetalization% of a thing of 40-75 mol is preferred, compatibility with a plasticizer falling that it is less than [40 mol %], and mixing a plasticizer of a complement to reservation of penetration resistance --- hard --- ** --- there is a case. If 75-mol % is exceeded, while a mechanical strength of an interlayer for glass laminates obtained will fall, in order to obtain resin, prolonged reaction time is required, and it is not sometimes desirable on a process. It is 60-75-mol % more preferably, if it is less than [60 mol %], since hygroscopicity becomes high, a white bluish mark will happen easily. It is 64-71-mol % preferably.

[0033]In the above-mentioned plasticized polyvinyl-acetal resin, the following [30 mol %] have a preferred vinyl acetate component. Since it will become easy to cause blocking at the time of manufacture of resin if 30-mol % is exceeded, it becomes difficult to manufacture. Preferably, it is less than 19 mol %.

[0034]The above-mentioned plasticized polyvinyl-acetal resin comprises a vinyl-acetal ingredient, a vinyl alcohol ingredient, and a vinyl acetate component, and each of these component amounts, it can measure based on, for example, a JIS K 6728 polyvinyl-butyral test method" and a nuclear magnetic resonance method (NMR).

[0035]When the above-mentioned polyvinyl-acetal resin is except polyvinyl butyral resin, a vinyl alcohol component amount and the amount of vinyl acetate components can be measured, and the remaining vinyl-acetal component amounts can be computed by deducting both the above-mentioned component amounts from 100.

[0036]The above-mentioned polyvinyl-acetal resin can be conventionally manufactured by a publicly known method, for example, solution obtained by dissolving polyvinyl alcohol in warm water --- a predetermined temperature --- for example, it holds at 10-20 ° preferably, and 0-95 % of acetalization reactions are advanced, adding and stirring a necessary acid catalyst and aldehyde. Subsequently, reaction temperature is raised to 70 °, it ripes, a reaction is completed, and a method of performing neutralization, rinsing, and desiccation and obtaining powder of polyvinyl-acetal resin, etc. are mentioned after that.

[0037]As polyvinyl alcohol used as the above-mentioned raw material, a thing of the average degrees of polymerization 500-5000 is preferred, and a thing of the average degrees of polymerization 1000-2500 is more preferred. When it is less than 500, the penetration resistance of a glass laminate obtained may fall, if 5000 is exceeded, shaping of a resin layer will carry out --- hard --- ** --- there are things and, moreover, intensity of a resin layer may become strong too much.

[0038]Since it is preferred to set a vinyl acetate component of polyvinyl-acetal resin obtained to less than 30 mol % therefore, a saponification degree of the above-mentioned polyvinyl alcohol has a preferred thing beyond 70 mol %. When it is less than [70 mol %], the transparency of resin and heat resistance may fall and reactivity may also fall. It is a thing beyond 95 mol % more preferably.

[0039]An average degree of polymerization and a saponification degree of the above-mentioned polyvinyl alcohol can be measured, for example based on JIS K 6728 "polyvinyl alcohol test-method." As the above-mentioned aldehyde, aldehyde of the carbon numbers 3-10 is preferred. The moldability of resin layers with a carbon number enough by less than three may not be obtained. If 10 is exceeded, the reactivity of acetalization will fall, and moreover it will become easy to generate a block of resin during a reaction, and will become easy to follow difficulty on composition of resin.

40]It is not limited especially as the above-mentioned aldehyde, but for example, propionaldehyde, acetaldehyde, isobutyraldehyde, a valerolaldehyde, aliphatic series, such as n-hexylaldehyde, 2-methylaldehyde, n-hexylaldehyde, n-octyl aldehyde, n-decyl aldehyde, n-undecyl aldehyde, n-dodecylaldehyde, aromatic series, aliphatic series, aliphatic series, etc. are mentioned. In particular, they are n-butylaldehyde of the carbon numbers 4-8, n-hexylaldehyde, 2-methylaldehyde, and n-octyl aldehyde. Since adhesive strength of each resin layer becomes high, and n-butylaldehyde of the carbon number 4 is excellent also in weatherability by use of vinyl-acetal resin obtained and moreover becomes easy [manufacture of resin], it is more preferred. These may be used alone and two or more sorts may be used together.

41]In an interlayer of this invention, it is preferred that particle diameter of sodium salt in an interlayer is 10 micrometers or less, and it is 5 micrometers or less preferably. About particle diameter of potassium salt in an interlayer, it is preferred that it is 10 micrometers or less, and it is 5 micrometers or less preferably. The finer moderate still more desirable one of particle diameter or 0 is good.

42]Since condensation of water may be promoted when particle diameter of the above-mentioned lithium salt exceeds 10 micrometers or particle diameter of potassium salt exceeds 10 micrometers, an become a big cause of a white bluish mark by moisture absorption of an interlayer obtained.

43]Although particle diameter of the above-mentioned sodium salt and particle diameter of potassium salt point out particle diameter in inside of an interlayer. Although particle diameter of lithium salt in polyvinyl-acetal resin which is the main raw material, and particle diameter of sodium salt may decrease in process of film production. As for particle diameter of sodium salt, particle diameter of potassium salt, also in polyvinyl-acetal resin, since particle diameter may be 1, it is preferred that it is in a mentioned range.

44]Particle diameter of sodium salt in the above-mentioned interlayer and potassium salt can be measured by imaging of a secondary ion image which used a time-of-flight type secondary-ion-scanning device (TOF-SIMS).

45]In an interlayer of this invention, it is preferred that sodium concentration is 50 ppm or less, but potassium concentration in an interlayer, it is preferred that it is 100 ppm or less. More preferably, sodium concentration is 0.5 ppm or more 15 ppm or less, and potassium concentration is 5 ppm or more 100 ppm or less.

46]Since it will grow up to be even a size by which a water molecule which gathered around a lithium element and a potassium element is visualized if a sodium content in the above-mentioned interlayer exceeds 50 ppm and potassium contents exceed 100 ppm, a white bluish mark may become variable. Preparing an interlayer less than 0.5 ppm in any [of a sodium content in the above-mentioned interlayer and potassium contents] case. Treatment which lengthens dramatically a process of washing a residual sodium element or a potassium element, or raises the degrees of washing to be used, such as water and raw material, by preparation of resin may be needed, great expense may be required, and it may not be desirable practically.

47]Sodium concentration and potassium concentration in the above-mentioned interlayer can be identified by ICP luminescence ultimate analysis. After the above-mentioned ICP luminescence ultimate analysis heats and decomposes a sample with sulfuric acid and nitric acid and sets the same of a decomposition product with ultrapure water, it is the method of quantifying by the ICP-MS method.

48]Mixing of the above-mentioned sodium and/or potassium. For example, in order to neutralize acid catalyst of sulfuric acid used for a reaction, chloride, etc. in preparation of polyvinyl-acetal resin, it originates in having used a neutralizer having contained a sodium element or potassium elements, such as sodium carbonate, sodium bicarbonate, sodium acetate, sodium hydroxide, sodium carbonate, potassium bicarbonate, potassium acetate, and a potassium hydrate.

49]In a manufacturing method of the above-mentioned polyvinyl-acetal resin, a neutralization process can prevent an acid catalyst like indispensable chloride (HCl) remaining in resin, and causing radiation of resin itself in a generation reaction of polyvinyl-acetal resin which is a process before.

50]As the above-mentioned neutralizer, alkali metal salt and alkaline earth metal salt can be used. In if alkaline-earth metals remain so much in an interlayer unlike an alkaline metal, they are preferred at a point which can control a white bluish mark under high humidity.

[0051]As the above-mentioned alkaline earth metal salt, calcium salt, such as barium salt, calcium hydroxide, such as magnesium salt, barium hydroxide, such as aerated water matter magnesium, magnesium hydroxide, and basic magnesium carbonate, etc. are mentioned, for example.

[0052]Mixing of the above-mentioned sodium and/or potassium. When adding sodium salt or potassium salt, such as carboxylic acid and octylic acid, etc. as an adhesive strength regulator, a sodium element or a potassium element contains in used water, raw material, especially polyvinyl alcohol, etc., and it happens also by a case where these remain in an interlayer.

[0053]For example, an alkaline metal contained in the above-mentioned pure water can be reduced to 1 ppm or less by using ion exchange water. On the other hand, an alkaline metal contained in polyvinyl alcohol originates in sodium acetate generated in a manufacturing process of a polyvinyl alcohol raw material when saponifying polyvinyl acetate, and content is usually 0.4 to 1.5 % of the weight.

[0054]Therefore, when content of sodium acetate uses a polyvinyl alcohol raw material which is 0.4 or less % of the weight, sodium elements with difficult washing contained in resin can be reduced, it is stabilized by washing strengthening etc., and a sodium element can be 50 ppm or less.

[0055]In a manufacturing method of the above-mentioned polyvinyl-acetal resin, it can also carry out by rinsing polyvinyl-acetal resin, without performing the above-mentioned neutralization process, until it becomes pH five or more, and also drying below 60 °C. By fully rinsing, alkali metal content leading to a white bluish mark of a resin layer obtained can be held down to below the specified quantity until it becomes pH five or more. While preventing resin deterioration by mixing of an alkaline metal, and remains of an acid catalyst by [of 60 °C or less] drying at low temperature comparatively, corrosion by acid of a dryer can be prevented. Although a conventional method may be sufficient as a drying method, especially its vacuum-drying method is efficient, and excellent.

[0056]In the above-mentioned washing process, it is preferred to wash with not less than 40 °C water. It is for raising washing efficiency by temperature of water used at the time of washing being not less than 40 °C paying attention to resin in a slurry swelling above 40 °C, and preventing resin deterioration by mixing of an alkaline metal, or remains of an acid catalyst. At the time of washing, by using 40-60 °C wash water preferably, resin in a slurry swells, and acid (HCl) incorporated into resin and its neutralized substance (alkaline metal inclusion) are flushed easily, and can raise washing efficiency by not less than 40 °C. If wash water is lower than 40 °C, resin will not fully swell but it will be hard to go up an effect. When wash water is more expensive than 60 °C, while softening of resin may take place, particles may coalesce, formation of a block may be seen and a thing of stable particle diameter may not be obtained. Improvement in a large effect cannot be expected as compared with 60 °C water, but it becomes useless also in energy.

[0057]In order to prevent mixing of the above-mentioned sodium and potassium, carry out the acetalization reaction of polyvinyl alcohol, a chloride catalyst, and the aldehyde, and it faces compounding polyvinyl-acetal resin, Polyvinyl-acetal resin can be obtained as a stop agent and a chloride remover of a reaction using epoxide, and a method of performing film formation can also be mentioned after that. It is expressed with following general formula (1) as the above-mentioned epoxide. [0058]



[0059]R¹ and R² express hydrogen or an alkyl group. n expresses the integer of 0-3. 1,4-epoxide, 1,3-epoxide, such as trimethylene oxide, a tetrahydrofuran, and tetrahydropyran, 1,5-epoxide besides 1,2-epoxide, etc. are mentioned --- these --- one sort --- or two or more sorts may be used together. Especially as epoxide, ethyleneoxide, propylene oxide, etc. are preferred.

[0060]As amount of the above-mentioned epoxide used, the effective dose which can perform stop of a reaction and chloride removal can be used. Degradation of resin by mixing of an alkaline metal and remains of an acid catalyst can be prevented by replacing the method of using the above-mentioned epoxide with the neutralizer of a chloride catalyst, and stopping an acetalization reaction using epoxide, and also removing chloride.

[0061]In this invention, as for an interlayer for glass laminates, in order to prevent a white bluish mark

ier high humidity more effectively, it is preferred that it is what adds a dispersing agent. By adding above-mentioned dispersing agent, a sodium compound, a potassium compound, etc. which exist polyvinyl-acetal resin and a plasticizer can be distributed, and particle diameter of these elements i be made small.

62)Amine which is compatible in organic acid, resin, and a plasticizer which are compatible in a pound which can form sodium salt and potassium salt, and a complex, resin, and a plasticizer as above-mentioned dispersing agent is mentioned.

63)The compound which can form the above-mentioned sodium salt and potassium salt, and a plex can control a white bluish mark of an interlayer for glass laminates obtained, even if the ve-mentioned polyvinyl-acetal resin absorbs moisture, in order to make it hard to approach in er by carrying out hydrophobing of the circumference of metal salt, such as sodium salt and ssium salt.

64)It is not limited especially as a compound which can form the above-mentioned sodium and ssium salt, and a complex. For example, ethylenediaminetetraacetic acid, salicylaldehyde, salicylic d, Salicylanilide, oxalic acid, α 1,10-phenanthroline, an acetylacetone, β -hydroxyquinoline, ethylglyoxime, 1,1'-cyclohexane 2 acetic acid, salicylaldehyde, a glycine, etc. are mentioned. These e be used alone and may use two or more sorts together.

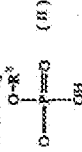
65)Although an addition of a compound which can form the above-mentioned sodium and ssium salt, and a complex is based on quantity of metal salt which remains to polyvinyl-acetal in, it is preferred that they are 0.02 - the amount part of duplex to the polyvinyl-acetal resin 100 ve-mentioned weight section. If a preventive effect of a white bluish mark by moisture absorption e become insufficient if it is less than 0.02 weight sections and the amount part of duplex is eeded, compatibility with the above-mentioned polyvinyl-acetal resin may become poor, and a elem may arise in transparency. It is 0.05 to 1 weight section more preferably.

66)As the above-mentioned dispersing agent, amine which is compatible in organic acid, resin, and lasticizer which are compatible in resin and a plasticizer can also be used. As organic acid which is patible in the above-mentioned resin and a plasticizer, polymer acid, such as monomer acid, such sulfonic acid, carboxylic acid, phosphoric acid, and nitric acid, polysulfone acid, and polycarboxylic d, etc. are mentioned, and sulfonic acid, carboxylic acid, and phosphoric acid are especially ferred. These may be used independently and may be used together two or more sorts.

67)It is using at least one sort more preferably from a group which consists of sulfonic acid whose bon numbers are 2-21, carboxylic acid whose carbon numbers are 2-20, and phosphoric acid whose ressed with following general formula (II) as organic acid which is compatible in the above- nioned resin and a plasticizer.

68)

rmula 2)



69)[R³ expresses among a formula the aliphatic series system hydrocarbon group whose carbon bers are 1-18, or the aromatic system hydrocarbon group whose carbon numbers are 1-18.) R⁴ resses a hydrogen atom, the aliphatic series system hydrocarbon group whose carbon numbers 1-18, or the aromatic system hydrocarbon group whose carbon numbers are 1-18.

70)In the sulfonic acid whose above-mentioned carbon numbers are 2-21, if a carbon number is e than two, When hydrophilic nature may become high, compatibility with polyvinyl-acetal resin e worsen, distributing may become insufficient and a carbon number exceeds 21, it becomes ropophobicity, compatibility with polyvinyl-acetal resin worsens, and there is a possibility of causing so separation. A carbon number is a thing of 7-19 more preferably.

71)As sulfonic acid which is 2-21, the above-mentioned carbon number can use the thing of an iatic series system, the thing of an aromatic system, etc. The above-mentioned carbon number is imited especially as sulfonic acid which is 2-21. For example, benzenesulfonic acid, naphthalene ic acid, alkyl sulfonic acid whose carbon numbers of an alkyl group are 2-21. The carbon her of an alkyl group is alkyl naphthalene sulfonic acid etc. which are 2-11, and the carbon ber of the alkylbenzene sulfonic acid which is 2-15, and an alkyl group specifically, P-

toluenesulfonic acid, dodecylbenzenesulfonic acid, camphor sulfonic acid, hydroxypropanesulfonic acid, mesitylene sulfonic acid, etc. are mentioned. These may be used alone and may use two or more sorts together.

[0072]As for an addition of sulfonic acid whose above-mentioned carbon numbers are 2-21, it is preferred that they are 0.01 - the amount part of duplex to the polyvinyl-acetal resin 100 above-mentioned weight section. If a preventive effect of a white bluish mark according that they are less than 0.01 weight sections to moisture absorption may become insufficient and exceeds the amount part of duplex, degradation of resin may be promoted or this sulfonic acid itself may cause a white bluish mark. It is 0.03 to 1 weight section more preferably.

[0073]In carboxylic acid of 2-20 the above-mentioned carbon number. When hydrophilic nature may become it high that a carbon number is less than two, compatibility with polyvinyl-acetal resin may worsen, distributing may become insufficient and a carbon number exceeds 20, it becomes hydrophobicity, compatibility with polyvinyl-acetal resin worsens, and there is a possibility of causing phase separation. A carbon number is a thing of 6-14 more preferably.

[0074]As carboxylic acid which is 2-20, the above-mentioned carbon number can use a thing of an aliphatic series system, a thing of an aromatic system, etc. It may be dicarboxylic acid. The above-mentioned carbon number is not limited especially as carboxylic acid which is 2-20. For example, acetic acid, propionic acid, butanoic acid, isobutyric acid, 2-ethylbutanoic acid, Octanoic acid, 2-ethylhexyl acid, lauric acid, myristic acid, stearic acid, oxalic acid, malonic acid, succinic acid, adipic acid, pimelic acid, sebacic acid, oleic acid, benzoic acid, toluic acid, naphthoic acid, 1,1'-cyclohexane 2 acetic acid, salicylic acid, etc. are mentioned. These may be used alone and may use two or more sorts together.

[0075]As for an addition of carboxylic acid whose above-mentioned carbon numbers are 2-20, it is preferred that it is 0.01 to 3 weight section to the polyvinyl-acetal resin 100 above-mentioned weight section. When a preventive effect of a white bluish mark according that they are less than 0.01 weight sections to moisture absorption may become insufficient and exceeds three weight sections, compatibility with resin becomes poor and transparency has a possibility of a problem arising or promoting degradation of resin. It is 0.05 to 1 weight section more preferably.

[0076]In R³ and R⁴ of phosphoric acid which are expressed with the above-mentioned general formula (II), when a carbon number of the above-mentioned aliphatic series system hydrocarbon group or the above-mentioned aromatic system hydrocarbon group exceeds 18, it may become hydrophobicity and compatibility with polyvinyl-acetal resin may worsen. Carbon numbers are 6-12 more preferably.

[0077]Especially as phosphoric acid which is the above-mentioned general formula (II) and is expressed, it is not limited, but can use phosphoric acid generally used, and specifically, For example, methylphosphoric acid, ethylphosphoric acid, propylphosphoric acid, isopropylphosphoric acid, Butyl phosphate, laurylphosphate, stearylphosphoric acid, 2-ethylhexyl phosphoric acid, Di(2-ethylhexyl) phosphoric acid, isodecyl phosphoric acid, phenylphosphoric acid, dimethylphosphoric acid, diethylphosphoric acid, diisopropylphosphoric acid, dioctylphosphoric acid, diphenylphosphoric acid, dibenzylphosphoric acid, etc. are mentioned. These may be used alone and may use two or more sorts together.

[0078]As for an addition of phosphoric acid expressed with the above-mentioned general formula (II), it is preferred that they are 0.01 - the amount part of duplex to the polyvinyl-acetal resin 100 above-mentioned weight section. If a preventive effect of a white bluish mark by moisture absorption may become insufficient when it is less than 0.01 weight sections, and the amount part of duplex is exceeded, degradation of resin may be promoted or this phosphoric acid itself may cause a white bluish mark. It is 0.03 to 1 weight section more preferably.

[0079]Organic acid which is compatible in the above-mentioned resin and a plasticizer is used together with amine which is compatible in resin and a plasticizer. What is expressed with following general formula (III) can be conveniently used for amine which is compatible in the above-mentioned resin and a plasticizer.

[0080]

[Formula 3]



81][R⁵, R⁶, and R⁷ are a hydrogen atom, an aliphatic series system hydrocarbon group whose bon numbers are 1-20, or an aromatic system hydrocarbon group whose carbon numbers are 1-20 and a formula, respectively.) R⁵, R⁶, and R⁷ may be the same, and may differ from each other.

82]When a carbon number of the above-mentioned aliphatic series system hydrocarbon group or above-mentioned aromatic system hydrocarbon group exceeds 20, it may become hydrophobicity compatibility with polyvinyl-acetal resin may worsen. As for any one of R⁵, R⁶, and R⁷, it is ferred that it is a long chain, and a hydrocarbon group and R⁷ R⁵ and whose R⁶ are a hydrogen m and the carbon numbers 1-2 are a hydrocarbon group of the carbon numbers 6-16 more ferably.

83]As amine expressed with the above-mentioned general formula (III). For example, they are nitioned by nitrogen-containing heterocyclic compounds, such as aromatic amines, such as 1, 2 l alkylamine of the 3rd class, and aniline, and pyridine, etc., and specifically, Methylamine, ylamine, propylamine, hexylamine, octylamine, Decyl amine, dodecyl amine, tetradecylamine, aniline, uidine. The 1st class amine, such as naphthylamine; Dimethylamine; diethylamine; Secondary ne, such as diisopropyl amine, diethyl amine, diisopropyl amine, and N-methylamine; Trimethylamine, iary amine, such as triethylamine, N,N-dimethylhexylamine, N,N-dimethyl octylamine, N,N- ethyldecyl amine, N,N-dimethyldecyl amine, N,N-dimethylbenzylamine, and pyridine, etc. are tioned. These may be used alone and may use two or more sorts together.

84]As for an addition of amine expressed with the above-mentioned general formula (III), when a bon number uses sulfonic acid which is 2-21 as organic acid which is compatible in resin and a sticizer, it is preferred that they are 0.01 - the amount part of duplex to polyvinyl-acetal resin i weight section. When a preventive effect of a white bluish mark by moisture absorption may come insufficient when it is less than 0.01 weight sections, and the amount part of duplex is eeded, compatibility with resin becomes poor, a problem may arise in transparency or an rlayer may color it. It is 0.02 to 1 weight section more preferably.

85]As for an addition of amine expressed with general formula (III), when a carbon number uses boxylic acid which is 2-20 as organic acid which is compatible in the above-mentioned resin and a sticizer, it is preferred that it is 0.01 to 3 weight section to polyvinyl-acetal resin 100 weight tion. When a preventive effect of a white bluish mark by moisture absorption may become efficient if it is less than 0.01 weight sections and three weight sections are exceeded, npatibility with resin becomes poor, a problem may arise in transparency or an interlayer may color ; It is 0.05 to 1 weight section more preferably.

86]As for an addition of amine expressed with general formula (III), when using phosphoric acid ressed with the above-mentioned general formula (II) as organic acid which is compatible in the ve-mentioned resin and a plasticizer, it is preferred that they are 0.01 - the amount part of iox to polyvinyl-acetal resin 100 weight section. When a preventive effect of a white bluish mark moisture absorption may become insufficient when it is less than 0.01 weight sections, and the ount part of duplex is exceeded, compatibility with resin becomes poor, a problem may arise in nsparency or an interlayer may color it. It is 0.05 to 1 weight section more preferably.

87]As for the above-mentioned organic acid or amine, it is preferred to choose molecular cture and a molecular weight suitably and to be used according to the degree of acetalization of above-mentioned polyvinyl-acetal resin or a kind of the above-mentioned plasticizer.

88]Amine which is compatible in organic acid, resin, and a plasticizer which are compatible in resin i a plasticizer which are used as the above-mentioned dispersing agent. Respectively it becomes such as sulfonfyl ion, a carboxyl ion, phosphoric acid ion, and AMMONIUM ion, it acts on the face of metal salt of particle state which exists in polyvinyl-acetal resin, and combines with a tal ion which constitutes this metal salt, and its counter ion, or [and / that metal salt which these combined by kneading resin at the time of film production is distributed in resin, and metal salt of tie state becomes small as the result] --- or it disappears. For this reason, even if local densation of water is controlled and polyvinyl-acetal resin absorbs moisture, a white bluish mark

of an interlayer for glass laminates obtained can be controlled. Among organic acid which is compatible in resin and a plasticizer which are used as the above-mentioned dispersing agent, especially in phosphoric acid, since a role of a coupling agent of resin and glass is also played, and it has the character in which a film and glass do not exfoliate easily even if it absorbs moisture, exfoliation by moisture absorption of a glass laminate periphery can be controlled.

[0089]As for an interlayer for glass laminates, in this invention, it is preferred to contain at least one sort chosen from a group which consists of alkali metal salt and alkaline earth metal salt as an adhesive strength regulator.

[0090]It is not limited especially as the above-mentioned alkali metal salt and alkaline earth metal salt, for example, salts, such as potassium, sodium, and magnesium, are mentioned. As the above-mentioned salt, inorganic acid, such as organic acid, chlorides, such as carboxylic acid, such as acetylic acid, hexylacetic, butanoic acid, acetic acid, and formic acid, and nitric acid, is mentioned.

[0091]As the above-mentioned alkali metal salt and alkaline earth metal salt, it is more preferred that they are alkali metal salt of organic acid of the carbon numbers 5-16 and the alkaline earth metal salt of organic acid of the carbon numbers 5-16. It is magnesium salt of carboxylic acid of the carbon numbers 6-10, or dicarboxylic acid preferably.

[0092]It is not limited especially as magnesium salt of the above-mentioned carboxylic acid or dicarboxylic acid. For example, 2-ethylbutanoic acid magnesium, valeric-acid magnesium, hexanoic acid magnesium, magnesium heptanoate, octanoic acid magnesium, magnesium nonanoate, decanoic acid magnesium, glutaric acid magnesium, adipic acid magnesium, etc. are mentioned.

[0093]Magnesium salt of carboxylic acid of the above-mentioned carbon numbers 6-10, or dicarboxylic acid. By existing in a form of a salt, without ionizing in a film, and drawing a water molecule near, it is thought that it is possible to stop adhesive strength between an interlayer and glass, and penetration-proof performance of a glass laminate obtained by this can be made good. Since it is distributed over a membrane surface at high concentration, without condensing in a film and an excessive white bluish mark at the time of moisture absorption is not caused while a small quantity shows an adhesive strength adjustment effect, it is desirable.

[0094]As for the above-mentioned alkali metal salt and alkaline earth metal salt, it is preferred that particle diameter is 3 micrometers or less, and they are 1 micrometer or less more preferably. Since it will grow up to be even a size by which a water molecule which gathered around alkali metal salt and/or alkaline earth metal salt is visualized if it exceeds 3 micrometers, a white bluish mark may become remarkable and may not be preferred.

[0095]It is not limited especially as a means for the above-mentioned particle diameter to be 3 micrometers or less. For example, even if it is a method of using a compound which is easy to dissolve in polyvinyl-acetal resin or a plasticizer as an adhesive strength regulator, and a compound which is hard to dissolve in polyvinyl-acetal resin or a plasticizer, A method of using what it is hard to condense in polyvinyl-acetal resin and a plasticizer, a method of using together a dispersing agent which distributes them, a compatibilizer, etc., etc. are mentioned.

[0096]As a compound which is easy to dissolve in the above-mentioned combination, polyvinyl butyral resin is used as polyvinyl-acetal resin, for example, in combination using triethylene glycol 2-ethyl butyrate as a plasticizer, organic acid salt, such as octanoic acid magnesium, neo decanoic acid magnesium, and adipic acid magnesium, etc. are mentioned, and these one sort or two sorts or more are used suitably.

[0097]As a potassium compound which is easy to dissolve in the above-mentioned combination, potassium salt of organic acid, such as octanoic acid potassium, neo decanoic acid potassium, and potassium stearate, etc. are mentioned, and these one sort or two sorts or more are used suitably. [0098]As a sodium compound which is easy to dissolve in the above-mentioned combination, sodium salt of organic acid, such as sodium octanoate, neo decanoic acid, and sodium stearate, etc. are mentioned, and these one sort or two sorts or more are used suitably.

[0099]As a compound which is during combination of what it is hard to dissolve in the above-mentioned combination, and is hard to condense, magnesium salt of inorganic acid, such as a magnesium chloride and a magnesium nitrate, etc. are mentioned, and these one sort or two sorts or more are used suitably, for example.

[0100]As a dispersing agent which may be distributed according to concomitant use, or a compatibilizer, a compound which is hard to dissolve in the above-mentioned combination, Although

limited in particular, long chain system organic acid, such as alcohols and octanoic acid, such as anol and octyl alcohol, and nonanoic acid, is mentioned, and these one sort or two sorts or more used suitably, for example.

[01A] Method of using a compound which itself tends to dissolve in polyvinyl-acetal resin or a sticizer also in various above-mentioned methods is the most preferred, and a method of using a compound which it ranks second and is hard to condense in polyvinyl-acetal resin and a plasticizer is preferred.

[02]As the above-mentioned alkali metal salt and alkaline earth metal salt, when using a diester tem compound as a plasticizer, it is preferred that it is what has an acid component and the icture of a diester system compound. Since it can distribute stably and uniformly and can exist in in by having a structure similar to an acid component of a diester system compound used as a sticizer, aging is not caused.

[03]As the above-mentioned plasticizer, triethylene glycol di-2-ethyl butyrate. When using (it also ig hereafter called "3GH") or a dihexyl horse mackerel peat (henceforth "DHA"), by making metal ; of the carbon number 5 or carboxylic acid of 6 contain as an adhesive strength regulator. Since a of adhesive strength of an interlayer and glass with the passage of time can be prevented and vention from a white bluish mark and prevention from a fall with the passage of time of adhesive ngth can be reconciled, it is desirable. When using triethylene glycol di-2-ethylhexanoate neceforth "3GO") as a plasticizer for same reason, it is preferred that metal salt of carboxylic acid he carbon numbers 6-8 contains. When using tetraethylene Glyco Lot's 2-ethylhexanoate neceforth "4GO") as a plasticizer, it is preferred that metal salt of the carbon number 6 or boxylic acid of 7 contains.

[04]In order to prevent hydrolysis by heat at the time of film production of polyvinyl-acetal resin sticized (above-mentioned 3) as much as possible, it compares with a plasticizer like triethylene ol di-heptanoate (3G7) or tetraethylene glycol di-heptanoate (4G7). It is preferred to use a side in type plasticizer like 3GH which cannot cause hydrolysis easily, 3GO, and 4GO, or a horse kleral peat system type plasticizer like DHA.

[05]Above-mentioned 3GH has a track record long as a plasticizer for interlayers, and has the antage of being hard to cause hydrolysis, as compared with 3G7 and 4G7 grade which an organic i ingredient of a raw material is a side chain type, and are straight chain types. For example as mpared with 3GH, since the above-mentioned 3GO or 4GO are high boiling points, they have the antage of being hard to vaporize at the time of film production or doubling processing.

[06]Above-mentioned 3GH, 3GO, 4GO, and DHA may use together with other plasticizers which y be used alone and mentioned later. Although a concomitant use rate in particular of above- ntioned 3GH, 3GO, 4GO and DHA, and other plasticizers is not limited, it is preferred that the unt of concomitant use of other plasticizers is less than 50% of the weight of a plasticizer of ve-mentioned 3GH, 3GO, 4GO, and DHA. Since the characteristic which above-mentioned 3GH, GO, 4GO, and DHA have can weaken with other plasticizers if it is 50 % of the weight or more, an set of an adhesive strength regulator used corresponding to these may not no longer be onstrated fully.

[07]Metal salt of carboxylic acid as an adhesive strength regulator used when setting a plasticizer he above-mentioned interlayer as a specific thing. Although not limited in particular, pentanoic i metal salt (carbon number 5), hexanoic acid (2-ethylbutanoic acid) metal salt (carbon number 6), tanoic acid metal salt (carbon number 7), octanoic acid metal salt (carbon number 8), etc. are ntioned, and these one sort or two sorts or more are suitably used according to a kind of the ve-mentioned plasticizer. The above-mentioned carboxylic acid may be a straight chain type, and y be a side chain type.

[08]The moisture resistance of an interlayer obtained may become insufficient in case of metal salt se carbon number of the above-mentioned carboxylic acid is too small, generating of althium y become large, and a fall preventive effect of adhesive strength of an interlayer and glass with passage of time may become insufficient conversely in case of metal salt whose carbon number arboxylic acid is too large.

[09]Metal salt of carboxylic acid as the above-mentioned adhesive strength regulator. Although it y be used independently, respectively, formic acid magnesium, magnesium acetate, A metal salt tem adhesive strength regulator of carboxylic acid of the carbon numbers 1-4 like magnesium

propionate and magnesium butanoate; it may be used together with other adhesive strength regulators, such as a denaturation silicone oil system adhesive strength regulator mentioned later.

[010]When adding the above-mentioned alkali metal salt and/or alkaline earth metal salt as an adhesive strength regulator, the addition has 0.01 to 0.2 preferred weight section to polyvinyl-acetal resin 100 weight section. Since an adhesive strength adjustment effect will be lost if it is less than 0.01 weight sections, penetration-proof performance of a glass laminate obtained may fall. If 0.2 weight sections are exceeded, while spoiling the transparency of a glass laminate produced by carrying out bleed out, there is a possibility that adhesive strength of an interlayer and glass may decline too much. It is 0.03 to 0.09 weight section more preferably.

[011]As for sodium concentration, since especially a white bluish mark happens easily when the above-mentioned alkali metal salt is sodium salt, being set to 50 ppm or less is preferred. As the potassium element concentration, since a white bluish mark happens easily also when the above-mentioned alkali metal salt is potassium salt, being set to 100 ppm or less is preferred.

[012]The above-mentioned alkali metal salt and/or alkaline earth metal salt, Sulfuric acid used i others / in a case of adding as the above-mentioned adhesive strength regulator] in a reaction of polyvinyl-acetal resin. The above-mentioned metal salt may have contained in various raw materials, water, etc. which were used in a case where it originates in having used alkali metal salt and alkaline earth metal salt as a neutralizer of an acid catalyst of chloride etc., and a reaction of polyvinyl-acetal resin. Alkali metal salt and alkaline earth metal salt as the above-mentioned neutralizer can be diverted to some other purpose as an adhesive strength regulator.

[013]An interlayer for glass laminates of this invention consists of a plastic resin film which consists of additive agents, such as an above-mentioned dispersing agent and an adhesive strength regulator, by the above-mentioned polyvinyl-acetal resin, a plasticizer, and necessarily.

[014]As a plasticizer used in this invention, phosphoric acid system plasticizers, such as organic ester system plasticizers, such as a publicly known plasticizer used for this kind of interlayer, for example, monobasic acid ester, and polybasic acid ester, an organic phosphorus acid system, an organic phosphorus acid system, etc. are used.

[015]In the above-mentioned monobasic acid ester, for example Triethylene glycol, Glycol system ester obtained by a reaction with organic acid, such as butanoic acid, isobutyric acid, caproic acid, 2-ethylbutanoic acid, heptanoic acid, n-octylic acid, 2-ethylhexyl acid, pelargonic acid (n-nonylic acid), and decylacid, is preferred. In addition, ester of tetraethylene glycol, tripropylene glycol, and the above-mentioned organic acid is also used.

[016]As the above-mentioned polybasic acid ester, ester of organic acid, such as adipic acid, sebacic acid, and azelaic acid, and straight chain shape of the carbon numbers 4-8 or branched state alcohol is preferred, for example.

[017]As an example of the above-mentioned organic ester system plasticizer. Triethylene glycol di-2-ethyl butyrate, triethylene glycol di-2-ethylhexoate, Triethylene glycol dicaprylate, triethylene glycol di-n-octate, Triethylene glycol di-n-HEPUTOETO, tetraethylene glycol di-n-HEPUTOETO, other dibutyl sebacate, dioctylazelaate, and a dibutylcarbitol horse mackerel peat are used suitably.

[018]In addition, ethylene glycol di-2-ethyl butyrate, 1,3-propylene glycol di-2-ethyl butyrate, 1,4-propylene glycol di-2-ethyl butyrate, 1,4-butyleneglycol di-2-ethyl butyrate, 1,2-butyleneglycol di-2-ethylhexoate, diethylene glycol di-2-ethyl butyrate, Diethylene glycol di-2-ethylhexoate, dipropylene glycol di-2-ethyl butyrate, Triethylene glycol di-2-ethylhexoate, tetraethylene glycol di-2-ethyl butyrate, diethylene glycol JKAPURIOETO, etc. are used as a plasticizer.

[019]As the above-mentioned phosphoric acid system plasticizer, triethoxyethyl phosphate, isodecyl phenyl phosphate, triisopropyl phosphite, etc. are preferred. It is preferred to blend a diester system compound which consists of dicarboxylic acid and monohydric alcohol, or consists of monocarboxylic acid and dihydric alcohol in the above-mentioned plasticizer.

[0120]As the above-mentioned amount of plasticizers, 20 to 70 weight section is preferred to polyvinyl-acetal resin 100 weight section, and it is 40 to 60 weight section more preferably. The penetration resistance of a glass laminate which will be obtained if it is less than 20 weight sections may fall, if 70 weight sections are exceeded, a plasticizer will carry out bleeding, optical strain may become large or the transparency of a resin layer and an adhesive property may fall.

[0121]In this invention, a publicly known additive agent currently used for interlayers for glass laminates, such as conversion silicone oil for controlling penetration resistance besides a dispersing

43]adding the average degree of polymerization 1700, and the degree% of polyvinyl alcohol 275g of solification of 98.9 mol to the preparation pure water 2890g of Example 1 (1) resin --- warming --- dissolved. Temperature control of the system of reaction was carried out to 12 **, 35% of chloride catalysts [201 g of] and the *n*-butylaldehyde 148g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, make a reaction mixture, superfluous water washes, and unreacted *n*-butylaldehyde is flushed. The sodium hydroxide solution which is a general-purpose neutralizer neutralized the chloride catalyst, and also white colored polyvinyl butyral resin was obtained through rinsing and desiccation for 2 hours with superfluous water. The vinyl-acetal ingredient (degree of acetalization) of this resin was 65.0-mol %, the vinyl acetate component was 1.1-mol %.

44][2] Manufacture above-mentioned polyvinyl-butylal-resin 100 weight section of an interlayer, or mixing triethylene-glycol-di-2-ethyl-butylate 40 weight section as a plasticizer and fully rying out melt kneading of this with a roll mill, with the press-forming machine, press forming was carried out for 30 minutes, and 150 ** of 0.76-mm-thick interlayers were obtained. The result of measuring the particle diameter of the sodium salt in an interlayer, and potassium salt by using of the secondary ion image using a time-of-flight type secondary-ion-mass-spectroscopy (ToF-SIMS) device (product [made by PHIEVANS] TFS-2000 type). The particle diameter of 1 monomer and potassium salt of the particle diameter of the sodium salt in an interlayer was less than 0.5 micrometer. The bottom was a result and 6 ppm in fixed quantity in ICP luminescence analysis about the sodium element content of this interlayer. After ICP luminescence analysis heats and decomposes a sample with sulfuric acid and nitric acid and sets the time of this decomposition product with ultrapure water, it is the method of quantifying by the AES method using an ICP-AES device (the "ICAP-575 type" by a Japanese jar rel ash company).

45][3] It inserts from both sides using the manufacture above-mentioned interlayer of a glass laminate with a transparent flat glass (30 cm by 30 cmx2.5 mm in thickness). After putting this in rubber bag and carrying out indirect desulfurization mind with the degree of vacuum of 200torr for minutes, it moved to 90 ** oven, deaerated, and the vacuum press was carried out, holding for 30 minutes at 60 more **. Thus, this adhesion was performed for the glass laminate by which preliminary adhesion was carried out for 20 minutes on pressure 12 kg/cm² and conditions with a temperature of 100 **, in air type autoclave, and the transparent glass laminate was obtained. About the obtained laminate, an adhesive examination (pan Menu examination) and the moisture-proof white-blush-test were done by the following methods.

46][A quality assessment (1) adhesive-property (pan Menu) examination glass laminate is neglected adjusted to -18 ***0.6 ** temperature, and this is struck with the hammer whose weight of a id is 0.45 kg, and it grinds until the particle diameter of glass is set to 6 mm or less. And the degree of exposure of the film after glass carries out partial avulsion is judged with the boundary plate which carried out grade attachment beforehand, as shown in Table 1. This is for the adhesive strength of a glass plate and an interlayer to judge whether it is in the predetermined range.

47]

ble 1]

剥離の露出度 (%)	パシメル値
1.00	0
2.0	1
3.0	2
4.0	3
5.0	4
6.0	5
7.0	6
8.0	7
2 以下	8

[0148][2] The moisture-proof white-blush-mark sex-test resin layer was cut into the size of 4x4 cm. it was immersed in the ion exchange water of ordinary temperature (23 **, and Hayas of 24 hours after was measured using the integral equation turbidity meter (made by Tokyo Denshoku Co., Ltd.). The result was shown in Table 2.

[0149]In preparation of resin of example 2 Example 1, washing and a washing process after putting in a neutralizer were changed in 1.5 hours, and it carried out like Example 1 except it. In this case, as for the sodium element content in the obtained interlayer, the particle diameter of 3 micrometers and potassium salt of the particle diameter of 13 ppm and sodium salt was less than 0.5 micrometer.

[0150]In preparation of resin of example 3 Example 1, it carried out like Example 1 except having changed washing and a washing process after putting in a neutralizer in 2.5 hours. In this case, as for the sodium element content in the obtained interlayer, the particle diameter of 0.5 micrometer and potassium salt of the particle diameter of 3 ppm and sodium salt was less than 0.5 micrometer.

[0151]In preparation of resin of example 4 Example 1, it carried out like Example 1 except having changed washing and a washing process after putting in a neutralizer in 3.5 hours. In this case, as for the sodium element content in the obtained interlayer, the particle diameter of less than 0.5 and potassium salt of the particle diameter of 0.9 ppm and sodium salt was less than 0.5 micrometer.

[0152]In preparation of resin of comparative example 1 Example 1, it carried out like Example 1 except having changed washing and a washing process after putting in a neutralizer in 1 hour. In this case, the particle diameter of 17 ppm and sodium salt of the sodium element content in the obtained interlayer was 6 micrometers.

[0153]In preparation of resin of comparative example 2 Example 1, it carried out like Example 1 except having changed washing and a washing process after putting in a neutralizer in 0.5 hour. The particle diameter of 35 ppm and sodium salt of the sodium element content in the obtained interlayer was 13 micrometers. The evaluation result of the above Examples 1-4 and the comparative examples 1 and 2 was shown in Table 2.

[0154]

[Table 2]

		乾燥率				比較例			
		1	2	3	4	1	2	3	4
中	アセチル-ブチル酸塩	4	3	4	4	3	4	3	4
	アセチル-ブチル酸塩 (mol %)	85.8	85.8	85.8	85.8	85.8	85.8	85.8	85.8
	平均分子量	1700	1700	1700	1700	1700	1700	1700	1700
間	ビニルアセチル-ブチル酸塩 (mol %)	1.1	1.1	1.1	1.1	1.1	1.1	1.1	1.1
	可塑剤 (重量%)	40	40	40	40	40	40	40	40
	ナトリウム濃度 (ppm)	6	13	8	8	8	8	8	8
際	ナトリウム濃度 (mol %)	1	3	0.5	0.5	0.5	0.5	0.5	0.5
	カリウム濃度 (ppm)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
	カリウム濃度 (mol %)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
際	含有ケイ素の含有率 (%)	8	8	8	8	8	8	8	8
	水素価 2.4 酸素価のヘイズ (%)	28	27	24	24	24	24	24	24
	検査結果	○	○	○	○	○	○	○	○

plete, the water of an excess (it is 30 times the amount to resin) washes, and unreacted n-glycidyl ether is flushed. The potassium hydroxide solution which is a general-purpose neutralizer neutralized the chloride catalyst, and also white powdered polyvinyl butyral resin was obtained through rinsing and deacetalization for 2 hours with superfluous water. The vinyl-acetal ingredient (degree of acetalization) of this resin was 65.0-mol %, and the vinyl acetate component was 1.1-mol %.

51[2] Manufacture above-mentioned polyvinyl-butylal-resin, 100 weight section of an interlayer, or mixing triethylene-glycol-di-2-ethyl-butylate 40 weight section as a plasticizer and fully mixing out melt kneading of this with a roll mill, with the press-forming machine, press forming was carried out for 30 minutes, and 150 ** of 0.76-mm-thick interlayers were obtained. The bottom was a salt and 23 ppm in fixed quantity in ICP luminescence ultimate analysis about the potassium content of this interlayer. The particle diameter of less than [0.5 micrometer] mum and potassium salt of the particle diameter of the sodium salt in an interlayer was less than 3 micrometers.

52[3] It inserts from both sides using the manufacture above-mentioned interlayer of a glass laminate with a transparent float glass (30 cm by 30 cmx2.5 mm in thickness). After putting this in a rubber bag and carrying out indirect desulfurization mind with the degree of vacuum of 200torr for minutes, it moved to 90 ** oven, deaerated, and the vacuum press was carried out, holding for 30 minutes at 80 more **. Thus, this adhesion was performed for the glass laminate by which preliminary adhesion was carried out for 20 minutes on pressure 12 kg/cm² and conditions with a temperature of : ** in air type autoclave, and the transparent glass laminate was obtained.

53[4] Preparation of resin of example 6 Example 5, it carried out like Example 5 except having aged washing and a washing process after putting in a neutralizer in 2.5 hours. In this case, as for potassium elemental content in the obtained interlayer, the particle diameter of 1 micrometer and sum salt of the particle diameter of 5 ppm and potassium salt was less than 0.5 micrometer.

60[4] Preparation of resin of example 7 Example 5, it carried out like Example 5 except having aged washing and a washing process after putting in a neutralizer in 3.5 hours. In this case, as for potassium elemental content in the obtained interlayer, the particle diameter of less than 0.5 micrometer and sodium salt of the particle diameter of 0.7 ppm and potassium salt was less than 0.5 micrometer.

61[4] Preparation of resin of comparative example 3 Example 5, it carried out like Example 5 except having changed washing and a washing process after putting in a neutralizer in 1 hour. In this case, the particle diameter of 104 ppm and potassium salt of the potassium elemental content in the obtained interlayer was 6 micrometers.

62[4] Preparation of resin of comparative example 4 Example 5, it carried out like Example 5 except having changed washing and a washing process after putting in a neutralizer in 0.5 hour. In this case, the particle diameter of 220 ppm and potassium salt of the potassium elemental content in the obtained interlayer was 8 micrometers.

63[4] About the glass laminate obtained by the above Examples 5-8 and comparative examples 3 and 4, an adhesive examination (pan Menu examination) and a humidity test are done by the same method as the above-mentioned, and the evaluation result is summarized in Table 3, and is shown.

64[

ble 3]

	実施例	比較例			
		5	6	7	8
アセトール塩基添加	4	4	4	4	4
アセトール塩基濃度 (mol %)	65.0	65.0	65.0	65.0	65.0
平均重合度	1700	1700	1700	1700	1700
ビニルアセタール成分 (mol %)	1.1	1.1	1.1	1.1	1.1
可塑剤量 (重量部)	40	40	40	40	40
カリウム濃度 (ppm)	23	5	0.7	104	220
カリウム塩径 (μm)	3	1	0.5	6	8
ナトリウム濃度 (ppm)	11.7	0.6	0.6	—	—
ナトリウム塩径 (μm)	0.5	0.5	0.5	—	—
溶わセガラスの接着性 (パシメン値)	8	8	8	8	7
水浸漬 2.4 時間後のヘイズ (%)	24	20	14	60	92
総合判定	○	○	○	×	×

[0165] The moisture resistance excellent in Examples 5-7 is shown so that clearly from the above-mentioned result.

[0166] Adding the average degree of polymerization 1700, and the degree of polyvinyl alcohol 275g of saponification of 98.9 mol to the preparation pure water 2890g of Example 8 (1) resin --- warming --- it dissolved. Temperature control of the system of reaction was carried out to 12 **, 35% of chloride catalysts [201 g of] and the n-butylaldehyde 149g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, make a reaction complete, superfluous water washes, and unreacted n-butylaldehyde is flushed. The sodium hydroxide solution which is a general-purpose neutralizer neutralized the chloride catalyst, and also white powdered polyvinyl butyral resin was obtained through rinsing and deacetalization with superfluous water.

The degree of acetalization of this resin was 65.0-mol %.

[0167] (2) Triethylene-glycol-di-2-ethyl-butylate 40 weight section was mixed with manufacture above-mentioned polyvinyl-butylal-resin 100 weight section of the interlayer as a plasticizer, press forming was carried out with the press-forming machine, and the interlayer was obtained. The bottom was a result and 13 ppm in fixed quantity in the ICP luminescence elemental analysis device about the sodium element content of this interlayer. The particle diameter of the sodium salt in an interlayer was 3 micrometers.

[0168] (3) pinching the manufacture above-mentioned interlayer of a glass laminate between the 2.5-mm-thick float glasses of two sheets, putting this in a rubber bag, and carrying out suction deaeration under decompression of -600mmHg in autoclave. Heat crimping was simultaneously performed for 20 minutes on condition of the temperature of 80 **, and pressure ² of 5kg/cm, and the glass laminate was produced.

[0169] In production of the glass laminate of example 9 Example 8, the glass laminate was produced like Example 8 except the temperature at the time of heat crimping having been 80 **. The particle diameter of the sodium salt in an interlayer was 3 micrometers.

[0170] In production of the glass laminate of example 10 Example 8, the glass laminate was produced like Example 8 except the temperature at the time of heat crimping having been 100 **. The particle diameter of the sodium salt in an interlayer was 3 micrometers.

[0171] In preparation of resin of comparative example 5 Example 8, except having changed washing and a washing process for a short time a little, it carried out like Example 8 and the glass laminate was produced. In this case, the particle diameter of 30 ppm and sodium salt of the sodium element content in the obtained interlayer was 11 micrometers.

72]About the glass laminate obtained by the above Examples 8-10 and comparative example 5, adhesive examination (can Maru examination) and the heat resistance test were done by the following methods. About the humidity test, it carried out like Example 1.

73]After cooling a valuation method (1) adhesive-property (can Maru) examination glass laminate is 20 ** for 2 hours, An automatic hammer ring device is equipped, the whole surface of a glass laminate is uniformly hit by a hammer head, the area of the glass piece adhering to the interlayer of a glass laminate is observed visually, and adhesive strength (can Maru value) is judged as contrasted in the grade sample shown in Table 1. The adhesive strength minimum is one point and the minimum of a grade sample is a ten-point method of ten points. Curved-surface shaping of the form is carried out with the curvature which is 50 mm in radius, the used automatic hammer ring device has a hammer head with a blow effective diameter of 5 mm and a weight of 240g, and the striking power of a hammer head can be adjusted with a spring screw.

74]Take out and observe the existence of foaming visually, after neglecting the obtained glass laminate under 130 ** atmosphere for 2 hours based on the heat resistance test. JIS R3205 "glass laminate." The result of the above Examples 8-10 and the comparative example 5 was summarized in Table 4, and was shown.

75]

ble 4]

	実施例		比較例	
	8	9	10	5
ポリウレタン濃度 (ppm)	13	13	13	30
ポリウレタン濃度 (単位)	3	3	3	11
おせガラスの接着性 (バランデル値)	1	5	8	5
浸透 2.4 時間後のバレイズ (%)	37	38	33	58
おせガラスの耐熱衝撃試験	なし	なし	なし	なし
おせガラスの耐熱衝撃試験 (バランデル値)	なし	なし	なし	なし
合判定	○	○	○	×

76]adding the average degree of polymerization 1700, and the degree% of polyvinyl alcohol 275g of ionification of 98.9 mol to the preparation pure water 2890g of Example 11 (1) resin --- warning --- dissolved. Temperature control of the system of reaction was carried out to 12 **, 35% of chloride catalysts [201 g of] and the n-butylaldehyde 148g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, the reaction was made to complete, the water of the excess (they are 30 times to resin) washed, and unreacted n-butylaldehyde was flushed. The pH value was 5.1 when pH of the system at this time was measured, and with the centering dehydrator and resin of water content was obtained 50%. White powdered vinyl butyral resin was obtained for this resin through the desiccation under the atmosphere of 60 mmHg. The degree of acetalization of this resin was 65.0-mol %.

77]Triethylene-glycol-di-2-ethyl-butyrate 40 weight section was mixed with manufacture above-mentioned polyvinyl-butyral-resin 100 weight section of the interlayer as a plasticizer, pressing was carried out with the press-forming machine, and the interlayer was obtained. The bottom was a result and 0.7 ppm in fixed quantity in the ICP luminescence elemental analysis device about sodium element content in this interlayer. The particle diameter of the sodium salt in an interlayer was less than 0.5 micrometer.

78]The resin 1g was put into the valuation method (1) heat resistance test usual test tube, it heated in a 150 ** oil bath for 60 minutes, and the existence of degradation of resin was observed. The result of the above Examples 8-10 and the comparative example 5 was summarized in Table 5, and was shown.

79]adding the average degree of polymerization 1700, and the degree% of polyvinyl alcohol 275g of ionification of 98.9 mol to the preparation pure water 2890g of Example 11 (1) resin --- warning --- dissolved. Temperature control of the system of reaction was carried out to 12 **, 35% of chloride catalysts [201 g of] and the n-butylaldehyde 148g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, the reaction was made to complete, the water of the excess (they are 30 times to resin) washed, and unreacted n-butylaldehyde was flushed. The pH value was 5.1 when pH of the system at this time was measured, and with the centering dehydrator and resin of water content was obtained 50%. White powdered vinyl butyral resin was obtained for this resin through the desiccation under the atmosphere of 60 mmHg. The degree of acetalization of this resin was 65.0-mol %.

77]Triethylene-glycol-di-2-ethyl-butyrate 40 weight section was mixed with manufacture above-mentioned polyvinyl-butyral-resin 100 weight section of the interlayer as a plasticizer, pressing was carried out with the press-forming machine, and the interlayer was obtained. The bottom was a result and 0.7 ppm in fixed quantity in the ICP luminescence elemental analysis device about sodium element content in this interlayer. The particle diameter of the sodium salt in an interlayer was less than 0.5 micrometer.

saponification of 98.9 mol to the preparation pure water 2890g of Example 12 (1) resin --- warning --- dissolved. Temperature control of the system of reaction was carried out to 12 **, 35% of chloride catalysts [201 g of] and the n-butylaldehyde 148g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, and the reaction was made to complete, the water of the excess (they are 30 times to resin) washed, unreacted n-butylaldehyde was flushed, and the chloride catalyst was neutralized using the sodium hydroxide solution which is a general-purpose neutralizer. Then, it rinsed with water with a temperature of 50 ** of an excess (they are 30 times to resin), and white powdered polyvinyl butyral resin was obtained through desiccation. The degree of acetalization of this resin was 65.0-mol %.

80]Triethylene-glycol-di-2-ethyl-butyrate 40 weight section was mixed with manufacture above-mentioned polyvinyl-butyral-resin 100 weight section of the interlayer as a plasticizer, pressing was carried out with the press-forming machine, and the interlayer was obtained. The bottom was a result and 10 ppm in fixed quantity in the ICP luminescence elemental analysis device about sodium element content in this interlayer. The particle diameter of the sodium salt in an interlayer was 3 micrometers.

81]The manufacture above-mentioned interlayer of a glass laminate between the 2.5-mm-thick float glasses of two sheets, putting this in a rubber bag, and carrying out suction desiccation under decompression of -600mmHg in autoclave. Heat crimping was simultaneously performed for 20 minutes on condition of the temperature of 60 **, and pressure 5 kg/cm², and the glass laminate was produced.

82]In example 13 Example 12, the interlayer was obtained like Example 12 except [all] having used water with a temperature of 60 ** by washing after neutralization. The degree of acetalization of this interlayer was 65.0-mol %. The bottom was a result and 15 ppm in fixed quantity in the ICP luminescence elemental analysis device about the sodium element content in this interlayer. The particle diameter of the sodium salt in an interlayer was 4 micrometers.

83]adding the average degree of polymerization 1700, and the degree% of polyvinyl alcohol 275g of saponification of 98.9 mol to the preparation pure water 2890g of Example 14 (1) resin --- warning --- dissolved. Temperature control of the system of reaction was carried out to 12 **, 35% of chloride catalysts [201 g of] and the n-butylaldehyde 148g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, make a reaction complete, superfluous water washes, and unreacted n-butylaldehyde is flushed. Magnesium hydroxide which is a general-purpose neutralizer neutralized the chloride catalyst, and also it rinsed with superfluous water, and white powdered polyvinyl butyral resin was obtained through desiccation.

84]Triethylene-glycol-di-2-ethyl-butyrate 40 weight section was mixed with manufacture above-mentioned polyvinyl-butyral-resin 100 weight section of the interlayer as a plasticizer, pressing was carried out with the press-forming machine, and the interlayer was obtained. The bottom was a result and 0.7 ppm in fixed quantity in the ICP luminescence elemental analysis device about sodium element content in this interlayer. The particle diameter of the sodium salt in an interlayer was less than 0.5 micrometer.

85]The manufacture above-mentioned interlayer of a glass laminate between the 2.5-mm-thick float glasses of two sheets, putting this in a rubber bag, and carrying out suction desiccation under decompression of -600mmHg in autoclave. Heat crimping was simultaneously performed for 20 minutes on condition of the temperature of 60 **, and pressure 5 kg/cm², and the glass laminate was produced.

86]About the glass laminate obtained in above Examples 11-14, the heat resistance test was done by the following methods. About the humidity test, it carried out like Example 1. However, Example 14 carried out only the humidity test.

87]The resin 1g was put into the valuation method (1) heat resistance test usual test tube, it heated in a 150 ** oil bath for 60 minutes, and the existence of degradation of resin was observed. The result of the above Examples 11-14 was summarized in Table 5, and was shown.

88]

[Table 5]

	実施例				
	1.5	1.6	1.7	1.8	1.9
ポリビニルアルコールの重量 ナトリウム含量 (重量%)	0.1	0.4	0.3	0.1	0.1
ナトリウム濃度 (ppm)	8	13	9	2	2
ナトリウム粒径 (μm)	0.6	2	0.5	0.5	0.5
乾燥性試験	乾燥 なし	乾燥 なし	乾燥 なし	乾燥 なし	乾燥 なし
水浸後 2.4 時間後のヘイズ (%)	19	35	11	10	10
総合判定	○	○	○	○	○

[0196] Adding the average degree of polymerization 1700, and the saponification degree% of polyvinyl alcohol 275g of 98.9 mol to the preparation pure water 2890g of example 19(1) polyvinyl-acetal resin — warming — it dissolved. Temperature control of the system of reaction was carried out to 12 **, 35% of the weight of chloride catalysts [201 g of] and the n-butylaldehyde 148g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, make a reaction complete, superfluous water washes, and unreacted n-butylaldehyde is flushed. The sodium hydroxide solution which is a general-purpose neutralizer neutralized the chloride catalyst, and while powdered polyvinyl butyral resin was obtained through rinsing and desiccation for 2 hours with still more superfluous water. The amount of 64-mol % and survival acetyl groups of the degree of formation of average butyral of this resin was 1-mol %.

[0197] (2) the polyvinyl butyral resin (average-degree-of-polymerization 1700 and degree % of 64 mol of formation of average butyral) obtained by the manufacture above of the interlayer for glass laminates 0.08 copy of octanoic acid magnesium is added as a plasticizer to amount of residual acetyl groups 1 mol % 100 copy as 40 copies of triethylene glycol di-2-ethyl butyrate, and an adhesive regulator. After fully carrying out melt kneading with a roll mill, press forming was carried out for 30 minutes at 150 ** using the press-forming machine, and the interlayer for glass laminates of 0.76 mm of average thickness was obtained. The particle diameter of 10 ppm and sodium salt of the sodium content in the obtained interlayer for glass laminates was 1 micrometer. It was 0.9 micrometer when the particle diameter of the magnesium salt which exists in an interlayer was measured using the time-of-flight type secondary-ion-mass-spectroscopy device (TOF-SIMS).

[0198] (3) The interlayer for glass laminates obtained by the manufacture above of the glass laminate. After having put from the both sides with the transparent float glass (30 cm by 30 cm \times 3 mm in thickness), putting this in in the rubber bag and carrying out indirect desulfurization mind with the degree of vacuum of 20 torr for 20 minutes, it moved to oven, deaerated, and the vacuum press was carried out, holding for 30 minutes at 90 micro **. Thus, this sticking by pressure was performed for the glass laminate by which preparative pressure arrival was carried out for 20 minutes on condition of the temperature of 135 **, and pressure 12 kg/cm² in air type autoclave, and the glass laminate was obtained.

[0199] In manufacture of the interlayer for example 20 glass laminates, the interlayer for glass laminates and the glass laminate were obtained like Example 19 instead of 0.08 copy of octanoic acid magnesium as an adhesive regulator except having added 0.09 copy of neo decanoic acid magnesium. It was 0.5 micrometer when the particle diameter of the magnesium salt which exists in the interlayer for glass laminates obtained above was measured like Example 19.

[0200] In manufacture of the interlayer for example 21 glass laminates, the interlayer for glass laminates and the glass laminate were obtained like Example 19 instead of 0.08 copy of octanoic acid magnesium as an adhesive regulator except having added 0.04 copy of magnesium chloride. It was 2 micrometers when the particle diameter of the magnesium salt which exists in the interlayer for glass laminates obtained above was measured like Example 19.

[0201] In manufacture of example 22 polyvinyl-acetal resin, as a neutralizer, instead of sodium hydroxide solution, the average degree of polymerization 1700, and% of the powdered amount white

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	実施例				
	1.1	1.2	1.3	1.4	1.5
ナトリウム濃度 (ppm)	0.7	19	15	0.7	0.7
ナトリウム粒径 (μm)	0.5	3	4	0.5	0.5
乾燥性試験	乾燥 なし	乾燥 なし	乾燥 なし	乾燥 なし	乾燥 なし
浸漬後 2.4 時間後のヘイズ (%)	13	32	20	24	24
総合判定	○	○	○	○	○

[0199] Adding the polyvinyl alcohol 275g of the average degree of polymerization 1700 and 0.1 % of the weight of the degree % and sodium acetate content of saponification of 98.9 mol to the preparation pure water 2890g of Example 15 (1) resin — warming — it dissolved. Temperature control of the system of reaction was carried out to 12 **, 35% of chloride catalysts [201 g of] and the n-butylaldehyde 148g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, make a reaction complete, superfluous water (they 30 times to resin) washes, and unreacted n-butylaldehyde is flushed. After neutralizing a chloride catalyst using sodium hydroxide which is a general-purpose neutralizer, superfluous water washed white powdered polyvinyl butyral resin was obtained through desiccation. The degree of acetalization of this resin was 65-mol %.

[0200] (2) Triethylene-glycol-di-2-ethyl-butylate 40 weight section was mixed with manufacture above-mentioned polyvinyl-butyl-resin 100 weight section of the interlayer as a plasticizer, pressing was carried out with the press-forming machine, and the interlayer was obtained. The bottom is a result and 8 ppm in fixed quantity in the ICP luminescence elemental analysis device about the lum element content in this interlayer.

[0201] The interlayer was obtained like Example 15 except having used the polyvinyl alcohol 275g of example 16 average degree of polymerization 1700 and 0.4 % of the weight of the degree % and sodium acetate content of saponification of 98.9 mol. The degree of acetalization of this interlayer was 65.0-mol %. The bottom was a result and 13 ppm in fixed quantity in the ICP luminescence elemental analysis device about the sodium element content in this interlayer.

[0202] Adding the polyvinyl alcohol 275g of the average degree of polymerization 1700 and 0.1 % of the weight of the degree % and sodium acetate content of saponification of 98.9 mol to the preparation pure water 2890g of Example 17 (1) resin — warming — it dissolved. Temperature control of the system of reaction was carried out to 12 **, 35% of chloride catalysts [201 g of] and the n-butylaldehyde 148g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, make a reaction complete, superfluous water (they 30 times to resin) washes, and unreacted n-butylaldehyde is flushed. After neutralizing a chloride catalyst using octanoic acid magnesium which is a neutralizer, superfluous water washed and white powdered polyvinyl butyral resin was obtained through desiccation. The degree of acetalization of this resin was 65-mol %.

[0203] (2) Triethylene-glycol-di-2-ethyl-butylate 40 weight section was mixed with manufacture above-mentioned polyvinyl-butyl-resin 100 weight section of the interlayer as a plasticizer, pressing was carried out with the press-forming machine, and the interlayer was obtained. The bottom is a result and 2 ppm in fixed quantity in the ICP luminescence elemental analysis device about the lum element content in this interlayer.

[0204] The interlayer was obtained like Example 17 except having used ethyleneoxide as a neutralizer in example 18 chloride catalyst. The degree of acetalization of this interlayer was 65-mol %. The bottom was a result and 2 ppm in fixed quantity in the ICP luminescence elemental analysis device about the sodium element content in this interlayer. Using the interlayer obtained in above-mentioned examples 15-19, the same heat resistance test as Example 11 and the same humidity test as example 1 were done, and the result was shown in Table 6.

95]
file 6]

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ガラス 粉砕 (重量部)	ジエチル アミン (重量部)	液体形成化合物		反応シロコ ンポイン (重量部)	水浸漬 4 時間 後のヘイズ (%)
		種類	重量部		
24	100	40	40	0.04	0.05
25	100	40	40	0.04	0.05
26	100	40	40	0.04	0.05
27	100	40	40	0.04	0.05
28	100	40	40	0.04	0.05
29	100	40	40	0.04	0.05
30	100	40	40	0.04	0.05
31	100	40	40	0.04	0.05
32	100	40	40	0.04	0.05
33	100	40	40	0.04	0.05
34	100	40	40	0.04	0.05
35	100	40	40	0.04	0.05
36	100	40	40	0.04	0.05
37	100	40	40	0.04	0.05
38	100	40	40	0.04	0.05
39	100	40	40	0.04	0.05
40	100	40	40	0.04	0.05
41	100	40	40	0.04	0.05
42	100	40	40	0.04	0.05
43	100	40	40	0.04	0.05
44	100	40	40	0.04	0.05
45	100	40	40	0.04	0.05
46	100	40	40	0.04	0.05
47	100	40	40	0.04	0.05
48	100	40	40	0.04	0.05
49	100	40	40	0.04	0.05
50	100	40	40	0.04	0.05
51	100	40	40	0.04	0.05
52	100	40	40	0.04	0.05
53	100	40	40	0.04	0.05
54	100	40	40	0.04	0.05
55	100	40	40	0.04	0.05
56	100	40	40	0.04	0.05
57	100	40	40	0.04	0.05
58	100	40	40	0.04	0.05
59	100	40	40	0.04	0.05
60	100	40	40	0.04	0.05
61	100	40	40	0.04	0.05
62	100	40	40	0.04	0.05
63	100	40	40	0.04	0.05
64	100	40	40	0.04	0.05
65	100	40	40	0.04	0.05
66	100	40	40	0.04	0.05
67	100	40	40	0.04	0.05
68	100	40	40	0.04	0.05
69	100	40	40	0.04	0.05
70	100	40	40	0.04	0.05
71	100	40	40	0.04	0.05
72	100	40	40	0.04	0.05
73	100	40	40	0.04	0.05
74	100	40	40	0.04	0.05
75	100	40	40	0.04	0.05
76	100	40	40	0.04	0.05
77	100	40	40	0.04	0.05
78	100	40	40	0.04	0.05
79	100	40	40	0.04	0.05
80	100	40	40	0.04	0.05
81	100	40	40	0.04	0.05
82	100	40	40	0.04	0.05
83	100	40	40	0.04	0.05
84	100	40	40	0.04	0.05
85	100	40	40	0.04	0.05
86	100	40	40	0.04	0.05
87	100	40	40	0.04	0.05
88	100	40	40	0.04	0.05
89	100	40	40	0.04	0.05
90	100	40	40	0.04	0.05
91	100	40	40	0.04	0.05
92	100	40	40	0.04	0.05
93	100	40	40	0.04	0.05
94	100	40	40	0.04	0.05
95	100	40	40	0.04	0.05
96	100	40	40	0.04	0.05
97	100	40	40	0.04	0.05
98	100	40	40	0.04	0.05
99	100	40	40	0.04	0.05
100	100	40	40	0.04	0.05

18]adding the average degree of polymerization 1700, and the degree% of polyvinyl alcohol 275g of
 onification of 98.9 mol to the preparation pure water 2890g of example 29(1) polyvinyl-acetal resin
 warming — it dissolved. Temperature control of the system of reaction was carried out to 12 **,
 g of 35-% of the weight chloride catalysts and the n-butylaldehyde 148g were added, this
 perature was held, and the reactant was deposited. With superfluous water, hold the system of
 ction at 45 ** for 3 hours, make a reaction complete, wash, and Then, unreacted n-butylaldehyde
 shout. The sodium hydroxide solution which is a general-purpose neutralizer neutralized the
 oride catalyst, and also white powdered polyvinyl butyral resin was obtained through rinsing and
 ication for 2 hours with superfluous water. The amount of 64-mol % and survival acetyl groups of
 degree of formation of average butyral of this resin was 1-mol %.

19](2) manufacture profit *** polyvinyl butyral resin (average-degree-of-polymerization 1700
 degree % of 64 mol of formation of average butyral) of the interlayer for glass laminates To
 out of residual acetyl groups 1 mol %100 copy, as a plasticizer 40 copies of triethylene glycol di-
 butyrate, And 0.13 copy of dimethyl octylamine is added as 0.75 copy of
 lcoylbenzenesulfonic acid, and amine as organic acid. After carrying out melt kneading enough with
 30 ml, press forming was carried out for 30 minutes at 150 **, using the press-forming machine,
 the interlayer for glass laminates of 0.75 mm of average thickness was obtained. It was 50 ppm
 in the sodium content of the obtained interlayer was measured by ICP emission spectrometry. It
 s 4 micrometers when the particle diameter of the sodium element which exists in an interlayer
 s measured using the time-of-flight type secondary-ion-mass-spectrometry device (TOF-SIMS)
 20](3) It inserts from both sides using the manufacture above-mentioned interlayer of a glass
 inate with a transparent float glass (30 cm by 30 cm3 mm in thickness). After putting this in in
 rubber bag and carrying out indirect desulfurization mind with the degree of vacuum of 200orr for
 minutes, it moved to 90 ** oven, deaerated, and the vacuum press was carried out, holding for 30
 utes at 80 more **. Thus, this adhesion was performed for the glass laminate by which preliminary
 ion was carried out for 20 minutes on condition of the temperature of 135 **, and pressure 12
 cm² in air type autoclave, and the glass laminate was obtained. About the obtained glass laminate
 rlayer, the humidity test was done like Example 1 and the result was shown in Table 9.

21]In manufacture of the interlayer for example 30 glass laminates, 0.30 copy of octanoic acid is
 led instead of 0.75 copy of dodecylbenzenesulfonic acid as organic acid. Except having added 0.35
 y of decyl amine instead of 0.13 copy of dimethyl octylamine as amine, the resin layer was
 ained like Example 29 and the evaluated result was shown in Table 9. It was 5 micrometers when
 particle diameter of the sodium element which exists in the obtained interlayer was measured like
 mple 29

22]In manufacture of the interlayer for example 31 glass laminates, except having added 0.20 copy
 3(2-ethylhexyl) phosphoric acid instead of 0.75 copy of dodecylbenzenesulfonic acid as organic
 d, the resin layer was obtained like Example 29 and the evaluated result was shown in Table 9. It
 s 2 micrometers when the particle diameter of the sodium element which exists in the obtained
 rlayer was measured like Example 29.

23]In manufacture of the interlayer for comparative example 10 glass laminates, except not having
 led the organic acid and amine of the dispersing agent, the resin layer was obtained like Example
 and the evaluated result was shown in Table 9. It was 20 micrometers when the particle diameter

of the sodium element which exists in the obtained interlayer was measured like Example 29.
 {0224}In manufacture of the interlayer for comparative example 11 glass laminates, except not having
 added the organic acid and amine of the dispersing agent, and having changed washing time from 2
 hours in 3 hours, the resin layer was obtained like Example 29 and the evaluated result was shown in
 Table 9. It was 30 ppm when the sodium content which exists in the obtained interlayer was
 measured like Example 29. It was 13 micrometers when the particle diameter of the sodium element
 was measured like Example 29.

{0225}

{Table 9}

	実施例				比較例
	20	30	31	10	11
アセター-小量添加量	4	4	4	4	4
平均ブチラール化度 (モル%)	64.0	64.0	64.0	64.0	64.0
残存アセチル基量 (モル%)	1.0	1.0	1.0	1.0	1.0
平均重合度	1700	1700	1700	1700	1700
可塑剤量 (重量部)	40	40	40	40	40
サトリウム添加量 (ppm)	50	50	50	50	50
サトリウム残量 (ppm)	4	5	2	20	13
水浸漬 2 時間後のヘイズ	28	28	28	92	72
総合判定	○	○	○	×	×

{0226}Example 32 composition / preparation (composition of polyvinyl butyral resin) ion-exchange-
 water 2890 weight section, It heated and dissolved in 95 **, having supplied degree% of polyvinyl
 alcohol of saponification of 99.2 mol 198 weight section (4.5 mol of vinyl alcohol considerable amount)
 to the reactor with an agitating device, and agitating it with the average degree of polymerization
 1700. After cooled this solution at 30 **, having added 35-% of the weight chloride 196 weight section
 (1.9 mol) and n-butylaldehyde 152 weight section (2.1 mol), lowering solution temperature
 subsequently to 2 **, holding this temperature and polyvinyl butyral resin's depositing, temperature
 up of the solution temperature was carried out to 30 **, and it was held for 5 hours. After holding,
 sodium bicarbonate 147 weight section (1.7 mol) was added, it neutralized, rinsing and desiccation
 were performed, and the degree% of polyvinyl butyral resin of butyral-izing of 65 mol was obtained. It
 was 50 ppm when the sodium content of the obtained polyvinyl butyral resin was measured by ICP
 emission spectrometry. The particle diameter of sodium salt was 12 micrometers.

{0227}(Production of a resin layer) Polyvinyl-butylal-resin 100 obtained weight section, Triethyleneglycol-di-2-ethyl-butylate 40 weight section, p-toluenesulfonic-acid 0.43 weight section, And press
 forming of the kneaded material produced by supplying hexylamine 0.23 weight section to a roll mill,
 and kneading it was carried out for 10 minutes on condition of 150 ** and 120 kg/cm² with the
 press-forming machine, and the 0.8-mm-thick resin layer was obtained. The obtained resin layer was
 used and the moisture-proof white-blush-mark sex test was done like Example 1. The result was
 shown in Table 10.

{0228}Instead of example 33 hexylamine 0.23 weight section, the resin layer was obtained and
 evaluated like Example 32 except having used tetradecylamine 0.49 weight section. The result was
 shown in Table 10.

{0229}Instead of example 34 p-toluenesulfonic-acid 0.43 weight section, the resin layer was obtained
 and evaluated like Example 32 except having used dodecylbenzenesulfonic acid 0.75 weight section.
 The result was shown in Table 10.

{0230}The resin layer was obtained and evaluated like Example 32 instead of example 35 p-
 toluenesulfonic-acid 0.43 weight section and hexylamine 0.23 weight section except having used

	スルホン酸		アミン		水溶性と乾燥後の収率 (%)
	種類	添加割合	R ¹	R ²	
1.2	P-10240A-酸	0.53	ヘキシル	H	0.33
3.3	P-10240A-酸	0.43	ヘキシル	H	0.43
3.4	P-10240A-酸	0.75	ヘキシル	H	0.23
3.5	P-10240A-酸	0.15	デシル	H	0.17
3.6	P-10240A-酸	0.75	デシル	H	0.18
3.7	P-10240A-酸	0.75	デシル	H	0.42
3.8	P-10240A-酸	0.75	デシル	H	0.35
3.9	P-10240A-酸	0.37	メチル	メチル	0.18
4.0	P-10240A-酸	0.75	メチル	メチル	0.36
4.1	P-10240A-酸	0.75	メチル	メチル	0.43
1.2	—	0	—	—	0
1.3	—	0	デシル	H	0.18
1.4	P-10240A-酸	0.43	—	—	0
1.5	デシルペンタメチルベンゼンスルホン酸ナトリウム	—	—	—	0.43
1.6	デシルトリメチルベンゼンスルホン酸ナトリウム	—	—	—	0.33

[0243]The polyvinyl butyral resin compounded in example 42 (preparation of polyvinyl butyral resin) Example 32 was rinsed and dried further, and the polyvinyl butyral resin which reduced the content of sodium salt was obtained. It was 20 ppm when the sodium content of the obtained polyvinyl butyral resin was measured by ICP emission spectrometry. The particle diameter of sodium salt was 3.5 micrometers.

[0244](Production of a resin layer) Polyvinyl-butylal-resin 100 obtained weight section. On the same conditions as Example 32, it kneaded, press forming of triethylene-glycol-di-2-ethyl-butylate 40 weight section, dodecylbenzenesulfonic acid 0.33 weight section, and the decyl amine 0.17 weight section was carried out, and the 0.8-mm-thick resin layer was obtained. About the obtained resin layer, the moisture-proof white-blush-mark sex test was done like Example 1. The result was shown in Table 11.

[0245]The resin layer was obtained and evaluated like Example 42 instead of example 43 dodecylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section except having used dodecylbenzenesulfonic acid 0.17 weight section and decyl amine 0.09 weight section. The result was shown in Table 11.

[0246]The resin layer was obtained and evaluated like Example 42 instead of example 44 dodecylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section except having used dodecylbenzenesulfonic acid 0.03 weight section and decyl amine 0.02 weight section. The result was shown in Table 11.

[0247]Instead of example 45 decyl amine 0.17 weight section, the resin layer was obtained and evaluated like Example 42 except having used N,N-dimethyl octylamine 0.17 weight section. The result was shown in Table 11.

[0248]Instead of example 46 dodecylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section, The resin layer was obtained and evaluated like Example 42 except having used dodecylbenzenesulfonic acid 0.17 weight section and N,N-dimethyl octylamine 0.09 weight section. The result was shown in Table 11.

[0249]Instead of example 47 dodecylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section, The resin layer was obtained and evaluated like Example 42 except having used dodecylbenzenesulfonic acid 0.03 weight section and N,N-dimethyl octylamine 0.02 weight section. The result was shown in Table 11.

[0250]Instead of example 48 dodecylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section, The resin layer was obtained and evaluated like Example 42 except having used dodecylbenzenesulfonic acid 0.30 weight section and N,N-dimethyldecyl amine 0.20 weight section. The result was shown in Table 11.

lecylbenzenesulfonic acid 0.15 weight section and decyl amine 0.07 weight section. The result was shown in Table 10.

31]The resin layer was obtained and evaluated like Example 32 instead of example 38 p-toluenesulfonic-acid 0.43 weight section and hexylamine 0.23 weight section except having used lecylbenzenesulfonic acid 0.75 weight section and decyl amine 0.36 weight section. The result was shown in Table 10.

32]The resin layer was obtained and evaluated like Example 32 instead of example 37 p-toluenesulfonic-acid 0.43 weight section and hexylamine 0.23 weight section except having used lecylbenzenesulfonic acid 0.75 weight section and dodecyl amine 0.42 weight section. The result was shown in Table 10.

33]The resin layer was obtained and evaluated like Example 32 instead of example 38 p-toluenesulfonic-acid 0.43 weight section and hexylamine 0.23 weight section except having used lecylbenzenesulfonic acid 0.75 weight section and N,N-diethyl amine 0.55 weight section. The result was shown in Table 10.

34]Instead of example 39 p-toluenesulfonic-acid 0.43 weight section and hexylamine 0.23 weight section, The resin layer was obtained and evaluated like Example 32 except having used lecylbenzenesulfonic acid 0.37 weight section and N,N-dimethyl octylamine 0.18 weight section. The result was shown in Table 10.

35]Instead of example 40 p-toluenesulfonic-acid 0.43 weight section and hexylamine 0.23 weight section, The resin layer was obtained and evaluated like Example 32 except having used lecylbenzenesulfonic acid 0.75 weight section and N,N-dimethyl octylamine 0.36 weight section. The result was shown in Table 10.

36]Instead of example 41 p-toluenesulfonic-acid 0.43 weight section and hexylamine 0.23 weight section, The resin layer was obtained and evaluated like Example 32 except having used lecylbenzenesulfonic acid 0.75 weight section and N,N-dimethyldecyl amine 0.49 weight section. The result was shown in Table 10.

37]The resin layer was obtained and evaluated like Example 32 except not having used comparative example 12 p-toluenesulfonic-acid 0.43 weight section and hexylamine 0.23 weight section. The result was shown in Table 10.

38]Comparative example 13 p-toluenesulfonic-acid 0.43 weight section was not used, but the in layer was obtained and evaluated like Example 32 instead of hexylamine 0.23 weight section except having used decyl amine 0.36 weight section. The result was shown in Table 10.

39]The resin layer was obtained and evaluated like Example 32 except not having used comparative example 14 hexylamine 0.23 weight section. The result was shown in Table 10.

40]The resin layer was obtained and evaluated like Example 32 instead of comparative example 15 p-toluenesulfonic-acid 0.43 weight section and hexylamine 0.23 weight section except having used lithium dodecylbenzenesulfonate 0.80 weight section. The result was shown in Table 10.

41]The resin layer was obtained and evaluated like Example 32 instead of comparative example 16 p-toluenesulfonic-acid 0.43 weight section and hexylamine 0.23 weight section except having used lecyl-trimethylammonium-chloride 0.33 weight section. The result was shown in Table 10.

42]ble 10]

51] Instead of example 49 dodecylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section, the resin layer was obtained and evaluated like Example 42 except having used tetracyclenylbenzenesulfonic acid 0.12 weight section and N,N-dimethyldodecyl amine 0.08 weight section. The result was shown in Table 11.

52] The resin layer was obtained and evaluated like Example 42 except not having used comparative example 17 dodecylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section. The result was shown in Table 11.

53] Comparative example 18 dodecylbenzenesulfonic-acid 0.33 weight section was not used, but resin layer was obtained and evaluated like Example 42 instead of decyl amine 0.17 weight section except having used decyl amine 0.36 weight section. The result was shown in Table 11.

54] The resin layer was obtained and evaluated like Example 42 instead of comparative example 19 tetracyclenylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section except having used dodecylbenzenesulfonic acid 0.30 weight section. The result was shown in Table 11.

55] The resin layer was obtained and evaluated like Example 42 instead of comparative example 20 tetracyclenylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section except having used sodium dodecylbenzenesulfonate 0.50 weight section. The result was shown in Table 11.

56] The resin layer was obtained and evaluated like Example 42 instead of comparative example 21 tetracyclenylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section except having used dodecyl-trimethylammonium-chloride 0.50 weight section. The result was shown in Table 11.

57]

ble 11]

例	スルホン酸		アミン				水溶液 2.4 時間後のヘイズ (%)
	種類	添加量	R ¹	R ²	R ³	添加量	
42	テトラシクロペンタジエン	0.33	デシル	H	H	0.17	1.8, 8
43	テトラシクロペンタジエン	0.17	デシル	H	H	0.33	1.2, 5
44	テトラシクロペンタジエン	0.33	デシル	H	H	0.02	1.7, 2
45	テトラシクロペンタジエン	0.33	メチル	メチル	メチル	0.17	1.8, 5
46	テトラシクロペンタジエン	0.17	メチル	メチル	メチル	0.09	1.1, 2
47	テトラシクロペンタジエン	0.33	メチル	メチル	メチル	0.02	1.8, 6
48	テトラシクロペンタジエン	0.33	メチル	メチル	メチル	0.20	1.8, 4
49	テトラシクロペンタジエン	0.12	メチル	メチル	メチル	0.08	1.7, 2
17	—	0	—	—	—	0	3.8, 4
18	—	0	デシル	H	H	0.36	3.9, 2
19	テトラシクロペンタジエン	0.33	—	—	—	0	検定劣化
20	テトラシクロペンタジエン	0.33	メチル	メチル	メチル	0.50	6.8, 4
21	テトラシクロペンタジエン	0.50	メチル	メチル	メチル	0.50	5.4, 7

58] Polyvinyl-butylal-resin 100 weight section compounded in example 50 Example 32. On the conditions as Example 32, it kneaded, press forming of triethylene-glycol-di-2-ethyl-butylate weight section, octanoic acid 0.30 weight section, and the decyl amine 0.35 weight section was tried out, and the 0.8-mm-thick resin layer was obtained. About the obtained resin layer, the moisture-proof white-blush-mark sex test was done like Example 1. The result was shown in Table 12.

59] Instead of example 51 decyl amine 0.35 weight section, the resin layer was obtained and evaluated like Example 50 except having used dodecyl amine 0.40 weight section. The result was shown in Table 12.

60] Instead of example 52 decyl amine 0.35 weight section, the resin layer was obtained and evaluated like Example 50 except having used tetradecylamine 0.45 weight section. The result was shown in Table 12.

61] The resin layer was obtained and evaluated like Example 50 instead of example 53 octanoic-acid 0.30 weight section and decyl amine 0.35 weight section except having used myristic acid 0.50

weight section and dodecyl amine 0.40 weight section. The result was shown in Table 12.

[0262] Instead of example 54 decyl amine 0.35 weight section, the resin layer was obtained and evaluated like Example 50 except having used N,N-dimethyldodecyl amine 0.45 weight section. The result was shown in Table 12.

[0263] The resin layer was obtained and evaluated like Example 50 instead of example 55 octanoic-acid 0.30 weight section and decyl amine 0.35 weight section except having used benzoic acid 0.30 weight section and dodecyl amine 0.40 weight section. The result was shown in Table 12.

[0264] The resin layer was obtained and evaluated like Example 50 except not having used comparative example 22 decyl amine 0.35 weight section. The result was shown in Table 12.

[0265]

Table 12]

例	カルボン酸		アミン				水溶液 2.4 時間後のヘイズ (%)
	種類	添加量	R ¹	R ²	R ³	添加量	
50	オクタジエン	0.33	デシル	H	H	0.35	2.3, 4
51	オクタジエン	0.33	デシル	H	H	0.40	2.4, 3
52	オクタジエン	0.33	デシル	H	H	0.45	2.3, 7
53	ミリスチン酸	0.50	デシル	H	H	0.40	2.3, 8
54	オクタジエン	0.33	デシル	メチル	メチル	0.45	2.9, 8
55	オクタジエン	0.33	デシル	H	H	0.40	2.7, 0
22	オクタジエン	0.33	—	—	—	0	7.3, 0

[0266] Polyvinyl-butylal-resin 100 weight section prepared in example 56 Example 42. On the same conditions as Example 32, it kneaded, press forming of triethylene-glycol-di-2-ethyl-butylate 40 weight section, JI (n-butyl) phosphoric acid 0.16 weight section, and the dodecyl amine 0.14 weight section was carried out, and the 0.8-mm-thick resin layer was obtained. About the obtained resin layer, the moisture-proof white-blush-mark sex test was done like Example 1. After manufacturing the glass laminate like Example 1 using the obtained resin layer and neglecting it for four weeks under the atmosphere of 50 ** and 95%RH, an adhesive (pan Meru 0-1) with remarkable exfoliation of glass was carried out, and the adhesive property of the glass peripheral part which absorbed moisture was investigated. When the periphery had a portion (pan Meru 0-1) with remarkable exfoliation of glass compared with the center section at this time, the width of this portion was measured and it was considered as exfoliation distance. The result was shown in Table 13.

[0267] The resin layer was obtained and evaluated like Example 56 instead of example 57 JI (n-butyl) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used JI (n-butyl) phosphoric acid 0.17 weight section and N,N-dimethyl octylamine 0.13 weight section. The result was shown in Table 13.

[0268] The resin layer was obtained and evaluated like Example 56 instead of example 58 JI (n-butyl) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used JI (n-ethylhexyl) phosphoric acid 0.19 weight section and dodecyl amine 0.11 weight section. The result was shown in Table 13.

[0269] The resin layer was obtained and evaluated like Example 56 instead of example 59 JI (n-butyl) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used JI (n-ethylhexyl) phosphoric acid 0.20 weight section and N,N-dimethyl octylamine 0.10 weight section. The result was shown in Table 13.

[0270] The resin layer was obtained and evaluated like Example 56 instead of example 60 JI (n-butyl) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used JI (n-dodecyl) phosphoric acid 0.20 weight section and dodecyl amine 0.10 weight section. The result was shown in Table 13.

[0271] The resin layer was obtained and evaluated like Example 56 instead of example 61 JI (n-butyl) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used JI (2-dodecyl)

sphoric acid 0.21 weight section and N,N-dimethyl octylamine 0.00 weight section. The result was shown in Table 13.

72) The resin layer was obtained and evaluated like Example 50 instead of example 62 JI (n-butyl) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used

triethylphosphoric acid 0.17 weight section and dodecyl amine 0.13 weight section. The result was shown in Table 13.

73) The resin layer was obtained and evaluated like Example 50 instead of comparative example 23 (n-butyl) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used no (n-dodecyl) sodium phosphate 0.30 weight section. The result was shown in Table 13.

74) The resin layer was obtained and evaluated like Example 50 instead of comparative example 24 (n-butyl) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used triethylphosphoric acid 0.17 weight section and dodecyl amine 0.13 weight section. The result was shown in Table 13.

75) The resin layer was obtained and evaluated like Example 50 instead of comparative example 23 (n-butyl) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used no (n-dodecyl) sodium phosphate 0.30 weight section. The result was shown in Table 13.

リン酸	アミン					式量と4糖当量の比(%)	例題図像(3000)			
	R ¹	R ²	添加濃度	R ³	R ⁴			添加濃度		
50	α-ブチル	α-ブチル	0.15	アセト	アセト	9.14	10.5	9		
51	α-ブチル	α-ブチル	0.17	アセト	メチル	0.13	17.2	3		
52	2-エチルヘキシル	2-エチルヘキシル	0.19	アセト	アセト	0.14	12.6	9		
53	2-エチルヘキシル	2-エチルヘキシル	0.20	メチル	メチル	0.10	14.6	0		
90	α-ドデシル	α-ドデシル	0.21	アセト	アセト	0.16	13.4	9		
91	α-ドデシル	α-ドデシル	0.23	アセト	メチル	0.09	18.6	0		
62	フェニル	フェニル	0.17	アセト	アセト	0.13	16.7	0		
83	α-ドデシルリン酸ナトリウム							0.08	57.6	9
24	ドデシルトリメチルアミンニエタノールナトリウム							0.13	5.7	11

length regulator contain.

[0298]The polyvinyl butyral resin (average-degree-of-polymerization 1650 and degree % of 67 mol of butyral-izing) which set sodium content of neutralizing salt (sodium chloride) to 10 ppm by washing pure water as example 73 polyvinyl-acetal resin. The interlayer for glass laminates and the glass laminate were obtained like Example 70 except having used 32 mol of amount % and survival vinyl alcohol component amount % of residual acetyl groups of 1 mol.

[0299]The interlayer for glass laminates and the glass laminate were obtained like Example 70 except having made metal salt of carboxylic acid as shown in Table 15 as Example 74 -- a 78 adhesive-length regulator contain.

[0300]In manufacture of the interlayer for comparative example 27 glass laminates, as metal salt of carboxylic acid, instead of 0.071 copy of magnesium 2-ethylbutanoate, the interlayer for glass laminates and the glass laminate were obtained like Example 70 except having added 0.04 copy of 10^{-4} mol of magnesium acetate (carbon number 2).

[0301]The performance of the glass laminates obtained by Examples 70-78 and the comparative example 27 was evaluated like Example 63. The result was as being shown in Table 15.

[0302]

可成例 (添加例)	カルボン酸の金属塩 (添加例)	添加量 ($\times 10^{-4}$ モル)		バインダー		水浸漬 24 時間後の、 Δ の (%)
		種類	濃度	初期	経時後 A	
70	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	5	4.2
71	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	5	4
72	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	4	3.8
73	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	5	2.1
74	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	2	3.7
75	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	3	3.3
76	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	2	2.9
77	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	2	3.2
78	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	3	3.9
79	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	5	9.2

[0303]The combination presentation of the interlayer for example 79 glass laminates --- as polyvinyl-butyl resin --- polyvinyl butyral resin (average-degree-of-polymerization 1720 and degree % of 66 of butyral-izing) 33 mol of amount % and survival vinyl alcohol component amount % of residual vinyl groups of 1 mol. As the sodium content of 20 ppm, 100 copies of particle diameter of 2 micrometers of neutralizing salt, and a plasticizer, 39 copies of tetraethylene Glycol 2-ethylhexanoate (430). The interlayer for glass laminates was obtained like Example 63 except having added it as 0.079 copy (2.8×10^{-4} mol) of magnesium 2-ethylbutanoate (carbon number 7), the violet ray absorbent, and the antioxidant as metal salt of carboxylic acid. The glass laminate was obtained like Example 63 using the interlayer for glass laminates obtained above.

[0304]The interlayer for glass laminates and the glass laminate were obtained like Example 79 except having made metal salt of carboxylic acid as shown in Table 16 as Example 80 -- an 82 adhesive-length regulator contain.

[0305]In manufacture of the interlayer for comparative example 28 glass laminates, as metal salt of carboxylic acid, instead of 0.079 copy of magnesium 2-ethylbutanoate, the interlayer for glass laminates and the glass laminate were obtained like Example 79 except having added 0.04 copy of 10^{-4} mol of magnesium acetate (carbon number 2).

[0306]The performance of the glass laminates obtained by Examples 79-82 and the comparative example 28 was evaluated like Example 63. The result was as being shown in Table 16.

[0307]

ble 16]

可成例 (添加例)	カルボン酸の金属塩 (添加例)	添加量 ($\times 10^{-4}$ モル)		バインダー		水浸漬 24 時間後の、 Δ の (%)
		種類	濃度	初期	経時後 A	
80	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	5	3.2
81	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	3	4.6
82	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	3	2.8
83	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	3	2.6
84	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	5	9.2
85	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	5	9.2

[0295]The content of neutralizing salt (sodium chloride) with sodium concentration by washing according to pure water considering the combination presentation of the interlayer for example 83 glass laminates as polyvinyl-acetal resin 20 ppm, the polyvinyl butyral resin (average-degree-of-polymerization 1650 and degree % of 68 mol of butyral-izing) made into 2 micrometers of particles of neutralizing salt Amount % of 1 mol of residual acetyl groups, residual vinyl alcohol component amount 31 mol %100 copy. As a plasticizer, as metal salt of 38 copies of dihexyl horse mackarel peats (DHA), and carboxylic acid 0.071 copy (2.8×10^{-4} mol) of magnesium 2-ethylbutanoate (carbon number 6).

The interlayer for glass laminates was obtained like Example 63 except having considered it as the ultraviolet ray absorbent and the antioxidant. The glass laminate was obtained like Example 63 using the interlayer for glass laminates obtained above.

[0299]The interlayer for glass laminates and the glass laminate were obtained like Example 83 except having made metal salt of carboxylic acid as shown in Table 17 as Example 84 and an 85 adhesive-strength regulator contain.

[0300]In manufacture of the interlayer for comparative example 29 glass laminates, as metal salt of carboxylic acid, instead of 0.071 copy of magnesium 2-ethylbutanoate, the interlayer for glass laminates and the glass laminate were obtained like Example 83 except having added 0.04 copy (2.8×10^{-4} mol) of magnesium acetate (carbon number 2).

[0301]The interlayer for glass laminates and the glass laminate were obtained like Example 83 except having made metal salt of carboxylic acid as shown in Table 17 as a comparative example 30 adhesive-strength regulator contain.

[0302]The performance of the glass laminate obtained by Examples 83-85 and the comparative examples 29 and 30 was evaluated like Example 63. The result was as being shown in Table 17.

[0303]

Table 17]

可成例 (添加例)	カルボン酸の金属塩 (添加例)	添加量 ($\times 10^{-4}$ モル)		バインダー		水浸漬 24 時間後の、 Δ の (%)
		種類	濃度	初期	経時後 A	
86	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	5	3.8
87	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	4	4.8
88	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	3	2.6
89	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	5	9.2
90	DHA (3.6)	2-ethylbutanoate	0.071 (2.8)	5	5	9.2

[0304]Polyvinyl-butyl resin 100 weight section prepared in example 86 Example 42. On the same conditions as Example 42, it kneaded, press forming of triethylene-glycol-di-2-ethyl-butylate 40 weight section, camphor sulfonic acid 0.056 weight section, and the N,N-dimethyl octylamine 0.044 weight section was carried out, and the 0.8-mm-thick resin layer was obtained. About the obtained resin layer, the moisture-proof white-blush-mark sex test was done like Example 1. The result was shown in Table 18.

[0305]Instead of example 87 camphor sulfonic acid 0.056 weight section and N,N-dimethyl octylamine 0.044 weight section, The resin layer was obtained and evaluated like Example 88 except having used hydroxypropenesulfonic acid 0.043 weight section and N,N-dimethyl octylamine 0.057 weight section. The result was shown in Table 18

[006] Instead of example 88 camphor sulfonic acid 0.056 weight section, the resin layer was obtained and evaluated like Example 86 except having used mesitylene sulfonic acid 0.056 weight section. The result was shown in Table 18.

[007] Instead of example 89 camphor sulfonic acid 0.056 weight section and N,N-dimethyl octylamine 44 weight section, the resin layer was obtained and evaluated like Example 86 except having used decylbenzenesulfonic acid 0.06 weight section and pyridine 0.02 weight section. The result was shown in Table 18.

[008] Instead of example 90 camphor sulfonic acid 0.056 weight section and N,N-dimethyl octylamine 44 weight section, the resin layer was obtained and evaluated like Example 86 except having used decylbenzenesulfonic acid 0.061 weight section and p-toluidine 0.039 weight section. The result was shown in Table 18.

[009] Instead of example 91 camphor sulfonic acid 0.056 weight section and N,N-dimethyl octylamine 44 weight section, the resin layer was obtained and evaluated like Example 86 except having used cyclohexane 2 aceto-acid 0.048 weight section and dodecyl amine 0.104 weight section. The result was shown in Table 18.

[010] Instead of example 92 camphor sulfonic acid 0.056 weight section and N,N-dimethyl octylamine 44 weight section, the resin layer was obtained and evaluated like Example 86 except having used lauryl acid 0.042 weight section and dodecyl amine 0.08 weight section. The result was shown in Table 18.

[011] Instead of comparative example 31 camphor sulfonic acid 0.056 weight section and N,N-dimethyl octylamine 0.044 weight section, the resin layer was obtained and evaluated like Example 86 except having used pyridine 0.1 weight section. The result was shown in Table 18.

[012] Instead of comparative example 32 camphor sulfonic acid 0.056 weight section and N,N-dimethyl octylamine 0.044 weight section, the resin layer was obtained and evaluated like Example 86 except having used salicylic acid 0.1 weight section. The result was shown in Table 18.

[013] Instead of comparative example 33 camphor sulfonic acid 0.056 weight section and N,N-dimethyl octylamine 0.044 weight section, the resin layer was obtained and evaluated like Example 86 except having used camphor sulfonic acid 0.1 weight section. The result was shown in Table 18.

[014] Instead of comparative example 34 camphor sulfonic acid 0.056 weight section and N,N-dimethyl octylamine 0.044 weight section, the resin layer was obtained and evaluated like Example 86 except having used pyridinium chloride 0.1 weight section. The result was shown in Table 18.

例	有機酸	添加量	アミン		水浸漬 2.4 時間後のヘイズ (%)
			種類	添加量	
87	樟腦スルホン酸	0.056	N,N-ジメチルオクチルアミン	0.044	25.7
88	メチルベンゼンスルホン酸	0.048	N,N-ジメチルオクチルアミン	0.057	29.0
89	デシルベンゼンスルホン酸	0.061	N,N-ジメチルオクチルアミン	0.044	20.4
90	シクロヘキサン-2-アセト酸	0.048	ドデシルアミン	0.104	21.4
91	1,1-ピロリジン	0.061	ドデシルアミン	0.039	19.8
92	ラウリン酸	0.042	ドデシルアミン	0.08	26.1
93	—	—	ドデシルアミン	0.1	46.2
94	トリメチルアミン	0.1	—	—	35.1
95	樟腦スルホン酸・トリウム	—	—	0.1	66.2
96	塩化ピロリジン	—	—	0.1	54.7

[015] Instead of example 100 weight section prepared in example 93 Example 42, Triethyleneglycol-di-2-ethylbutyrate 40 weight section, octanoic acid 0.4 weight section, On the same conditions as Example 42, it kneaded, press forming of N,N-dimethyl octylamine 0.11 weight section [the 2-ethylbutanoic acid magnesium 0.037 weight section was carried out, and the 0.8-mm-thick

resin layer was obtained. About the obtained resin layer, the moisture-proof white-blush-mark sex test was done like Example 1.

[031] The friction test was done by the following method about the glass laminate obtained by fastening the above-mentioned resin layer between the glass plates (4x4 cm) of two sheets, and obtaining a glass laminate. The obtained laminate result was shown in Table 19.

[0318] (Friction test) After the glass laminate was immersed in underwater [with a temperature of 80 ± 1 °C for one week, it dried in oven with a temperature of 80 ± 1 °C for 4 hours. After repeating the process of this underwater immersion and oven drying 3 times, the existence of exfoliation of the interlayer in the circumference of a glass laminate was checked visually.

[0319] The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19 except having changed the quantity of example 94 N,N-dimethyl octylamine into 0.28 weight sections.

[0320] The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19 except having changed the quantity of example 95 octanoic acid into 0.1 weight sections, and having changed the quantity of N,N-dimethyl octylamine into 0.08 weight sections, respectively.

[0321] Change the quantity of example 96 octanoic acid into 0.2 weight sections, and the quantity of N,N-dimethyl octylamine is changed into 0.09 weight sections, respectively. The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19 except having used 2-ethylhexanoic acid magnesium 0.045 weight section instead of 2-ethylbutanoic acid magnesium 0.037 weight section.

[0322] Change the quantity of example 97 octanoic acid into 0.1 weight sections, and the quantity of N,N-dimethyl octylamine is changed into 0.06 weight sections, respectively. The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19 except having used 2-ethylhexanoic acid magnesium 0.045 weight section instead of 2-ethylbutanoic acid magnesium 0.037 weight section.

[0323] Except having changed the quantity of example 98 octanoic acid into 0.1 weight sections, and having used decyl amine 0.06 weight section and 2-ethylhexanoic acid magnesium 0.045 weight section instead of N,N-dimethyl octylamine 0.11 weight section and 2-ethylbutanoic acid magnesium 0.037 weight section. The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19.

[0324] Except having used di(2-ethoxy) phosphoric acid 0.03 weight section instead of example 99 octanoic acid 0.4 weight section, and having changed the quantity of N,N-dimethyl octylamine into 0.02 weight sections. The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19.

[0325] Using the glass laminate obtained by the comparative example 35 comparative example 12, the result obtained by doing only a friction test like Example 93 was looked like [Table 19], and was shown.

[0326] The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19 except not having used comparative example 36 octanoic acid and N,N-dimethyl octylamine.

[0327] The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19 except not having used comparative example 37 N,N-dimethyl octylamine.

[0328] The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19 except not having used comparative example 38 octanoic acid.

[0329] Comparative example 39 octanoic acid and N,N-dimethyl octylamine are not used. The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19 except having used 2-ethylhexanoic acid magnesium 0.045 weight section instead of 2-ethylbutanoic acid magnesium 0.037 weight section.

[0330] Instead of comparative example 40 octanoic acid 0.4 weight section, N,N-dimethyl octylamine 0.11 weight section, and 2-ethylbutanoic acid magnesium 0.037 weight section. The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19 except having used octanoic acid 0.2 weight section and 2-ethylhexanoic acid magnesium

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ATTEN AMENDMENT

ing date]May 13, Heisei 11

endment 1]

ment to be Amended]Specification

in(e) to be Amended]Claim

ethod of Amendment]Change

posed Amendment]

am(s)]

am 1]An interlayer for glass laminates when it is an interlayer for glass laminates which consists
of plasticized polyvinyl polyvinylacetate resin films and said 0.3-0.8-mm-thick interlayer is immersed in
water, wherein Hayes of 24 hours after is 50% or less.

am 2]The interlayer for glass laminates according to claim 1 whose particle diameter of sodium
salt in an interlayer is 10 micrometers or less.

am 3]The interlayer for glass laminates according to claim 1 or 2 whose particle diameter of
sodium salt in an interlayer is 5 micrometers or less.

am 4]The interlayer for glass laminates according to claim 1, 2, or 3 whose sodium concentration
in interlayer is 50 ppm or less.

am 5]The interlayer for glass laminates according to claim 1 whose particle diameter of potassium
salt in an interlayer is 10 micrometers or less.

am 6]The interlayer for glass laminates according to claim 1 or 5 whose particle diameter of
potassium salt in an interlayer is 5 micrometers or less.

am 7]The interlayer for glass laminates according to claim 1, 5, or 6 whose potassium
concentration in an interlayer is 100 ppm or less.

am 8]The interlayer for glass laminates according to claim 1, 2, 3, 4, 5, 6, or 7 which is a thing
containing a compound which can form sodium salt and potassium salt, and a complex.

am 9]The interlayer for glass laminates according to claim 1, 2, 3, 4, 5, 6, or 7 which is a thing
containing amine which is compatible in organic acid which is compatible in resin and a plasticizer.

in, and a plasticizer.

am 10]The interlayer for glass laminates according to claim 1, 2, 3, 4, 5, 6, 7, 8, or 9 which is a
thing containing at least one sort chosen from a group which consists of alkali metal salt and alkaline
earth metal salt.

am 11]The interlayer for glass laminates according to claim 10 whose alkali metal salt is a thing
whose particle diameter of 3 micrometers or less and whose alkaline earth metal salt is a thing with a
particle diameter of 3 micrometers or less.

am 12]The interlayer for glass laminates according to claim 10 or 11 whose alkali metal salt is the
alkali metal salt of organic acid of the carbon numbers 5-16 and whose alkaline earth metal salt is the
alkaline earth metal salt of organic acid of the carbon numbers 5-16.

am 13]A glass laminate characterized by making the interlayer for glass laminates according to
claim 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, or 12 come to intervene between glass of a couple at least.

am 14]An interlayer for glass laminates whose particle diameter of sodium salt in an interlayer is
less than 10 micrometers or less, and whose particle diameter of potassium salt in an interlayer is
less than 10 micrometers or less.

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[Claim 15]An interlayer for glass laminates whose particle diameter of sodium salt in an interlayer it is
an interlayer for glass laminates which consists of plasticized polyvinyl polyvinylacetate resin films, and
is 5 micrometers or less.

[Claim 16]The interlayer for glass laminates according to claim 14 or 15 whose sodium concentration
in an interlayer is 50 ppm or less.

[Claim 17]An interlayer for glass laminates whose sodium concentration in an interlayer it is an
interlayer for glass laminates which consists of plasticized polyvinyl polyvinylacetate resin films, and is
50 ppm or less.

[Claim 18]An interlayer for glass laminates whose particle diameter of potassium salt in an interlayer
it is an interlayer for glass laminates which consists of plasticized polyvinyl polyvinylacetate resin films,
and is 5 micrometers or less.

[Claim 19]An interlayer for glass laminates whose potassium concentration in an interlayer it is an
interlayer for glass laminates which consists of plasticized polyvinyl polyvinylacetate resin films, and is
100 ppm or less.

[Claim 20]An interlayer for glass laminates whose potassium concentration in an interlayer it is an
interlayer for glass laminates which consists of plasticized polyvinyl polyvinylacetate resin films,
particle diameter of potassium salt in an interlayer is 5 micrometers or less, and is 100 ppm or less.

[Claim 21]An interlayer for glass laminates whose potassium concentration in an interlayer it is an
interlayer for glass laminates which consists of plasticized polyvinyl polyvinylacetate resin films, sodium
concentration in an interlayer is 50 ppm or less, and is 100 ppm or less.

[Claim 22]The interlayer for glass laminates according to claim 14, 15, 16, 17, 18, 19, 20, or 21 which
is a thing containing a compound which can form sodium salt and potassium salt, and a complex.

[Claim 23]The interlayer for glass laminates according to claim 14, 15, 16, 17, 18, 19, 20, or 21 which
is a thing containing amine which is compatible in organic acid which is compatible in resin and a
plasticizer, resin, and a plasticizer.

[Claim 24]The interlayer for glass laminates according to claim 14, 15, 16, 17, 18, 19, 20, 21, 22, or 23
which is a thing containing at least one sort chosen from a group which consists of alkali metal salt
and alkaline earth metal salt.

[Claim 25]The interlayer for glass laminates according to claim 24 whose alkali metal salt is a thing
with a particle diameter of 3 micrometers or less and whose alkaline earth metal salt is a thing with a
particle diameter of 3 micrometers or less.

[Claim 26]The interlayer for glass laminates according to claim 24 or 25 whose alkali metal salt is the
alkali metal salt of organic acid of the carbon numbers 5-16 and whose alkaline earth metal salt is the
alkaline earth metal salt of organic acid of the carbon numbers 5-16.

[Claim 27]A glass laminate making claims 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24, and 25 or an
interlayer for glass laminates given in 26 come to intervene between glass of a couple at least.

[Translation done.]

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